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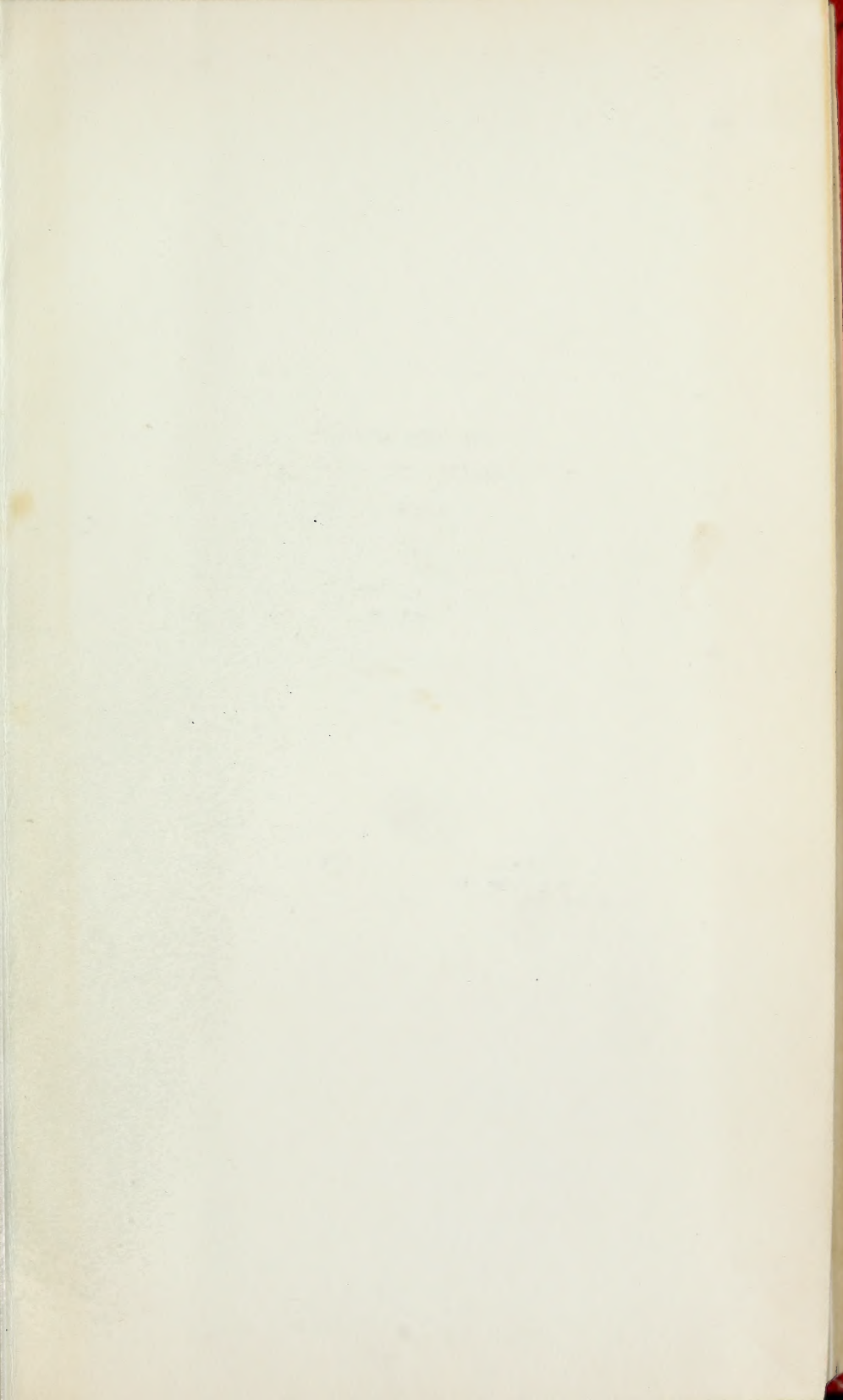
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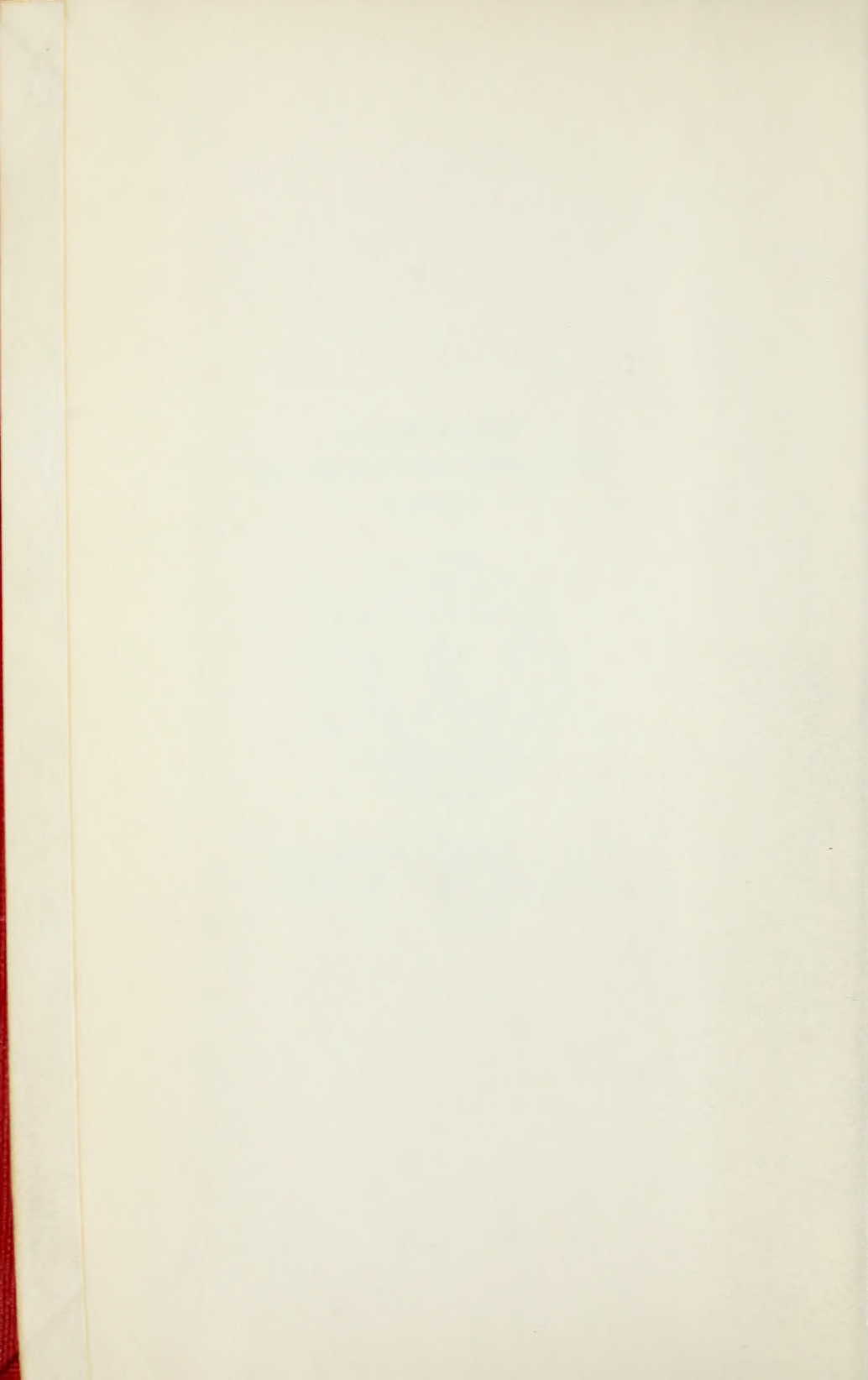
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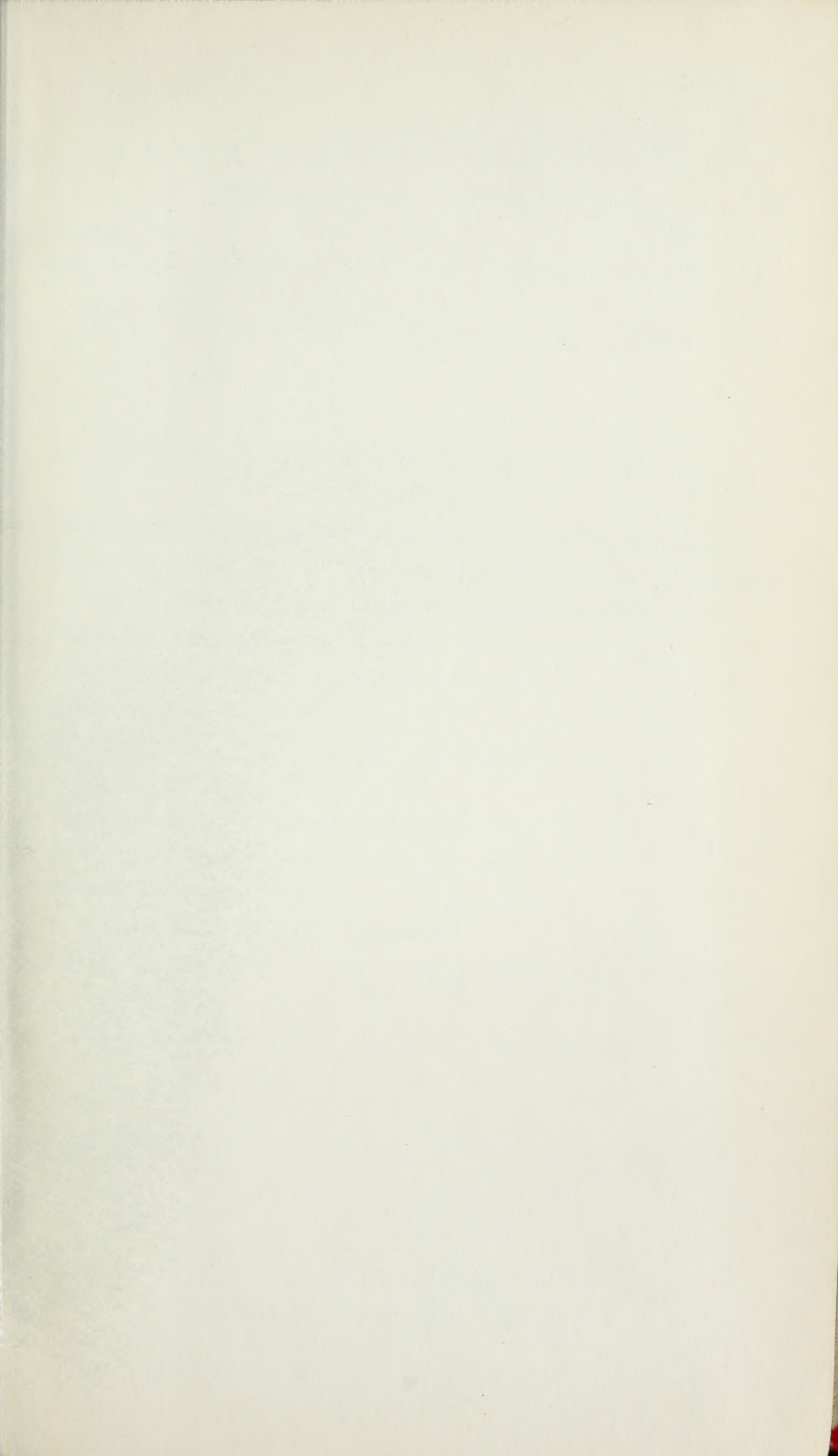


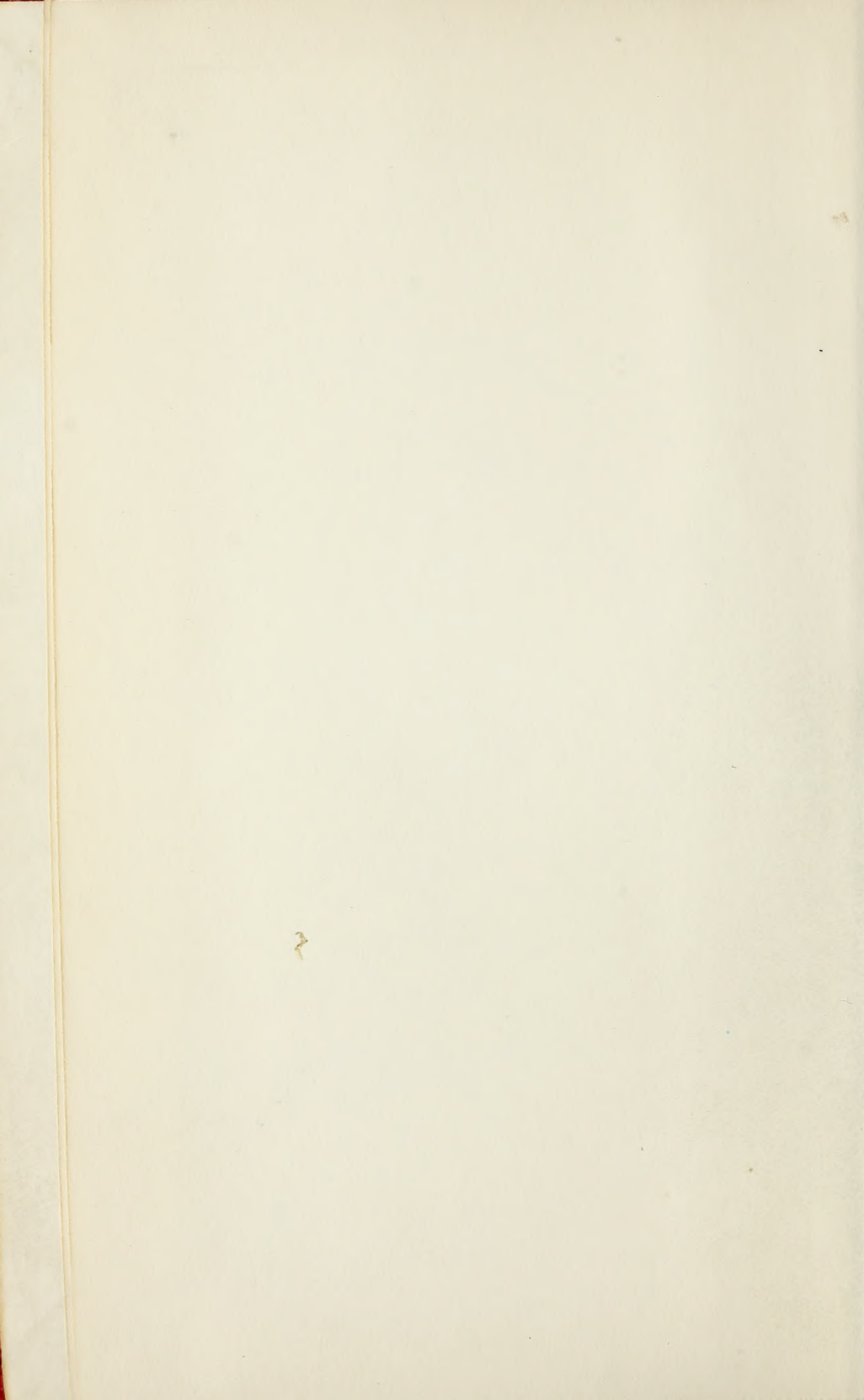
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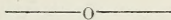








ERRATA. CHEMICAL BULLETIN No. 13 PART 1.



- p. 32 in composition of pure butter fat read, and palmitin, after, stearin.
- p. 34 Foot note, read, Ann. d. Chim et d. Phys. (Series V, Vol. 12, p. 469.)
- p. 39 read Sendtner and Hilger instead of Leudtner and Hilger.
- p. 55 8th line from bottom read 2.5 instead of 25 grams.
- p. 58 9th line from bottom read 25cc instead of 2½cc
- p. 59 8th line from top read 20 instead of 17.
- p. 60 11th line from bottom read 93 to 95½ instead of 95½
- p. 63 Second foot note, read volume 253.
- p. 70 Second half of 2nd equation read $C_3 H_2 + 3C_n H_{2n} O_2$
- p. 77 Foot note, read, appeared, instead of proved.
- p. 81 read for divisor in formula, $V - \frac{AS}{SI}$, instead of $V - S'$
(A equals grams fat in 100 grams milk.)
- p. 100 In formula (1) read + after f and read 2.665 instead of 2,665
 In formula (2) read t after 0.833.
 N. B. Please insert above corrections in ink in the text.

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U. S. DEPARTMENT OF AGRICULTURE.

DIVISION OF CHEMISTRY.

BULLETIN

No. 13.

FOODS

AND

FOOD ADULTERANTS.

BY DIRECTION OF

THE COMMISSIONER OF AGRICULTURE.

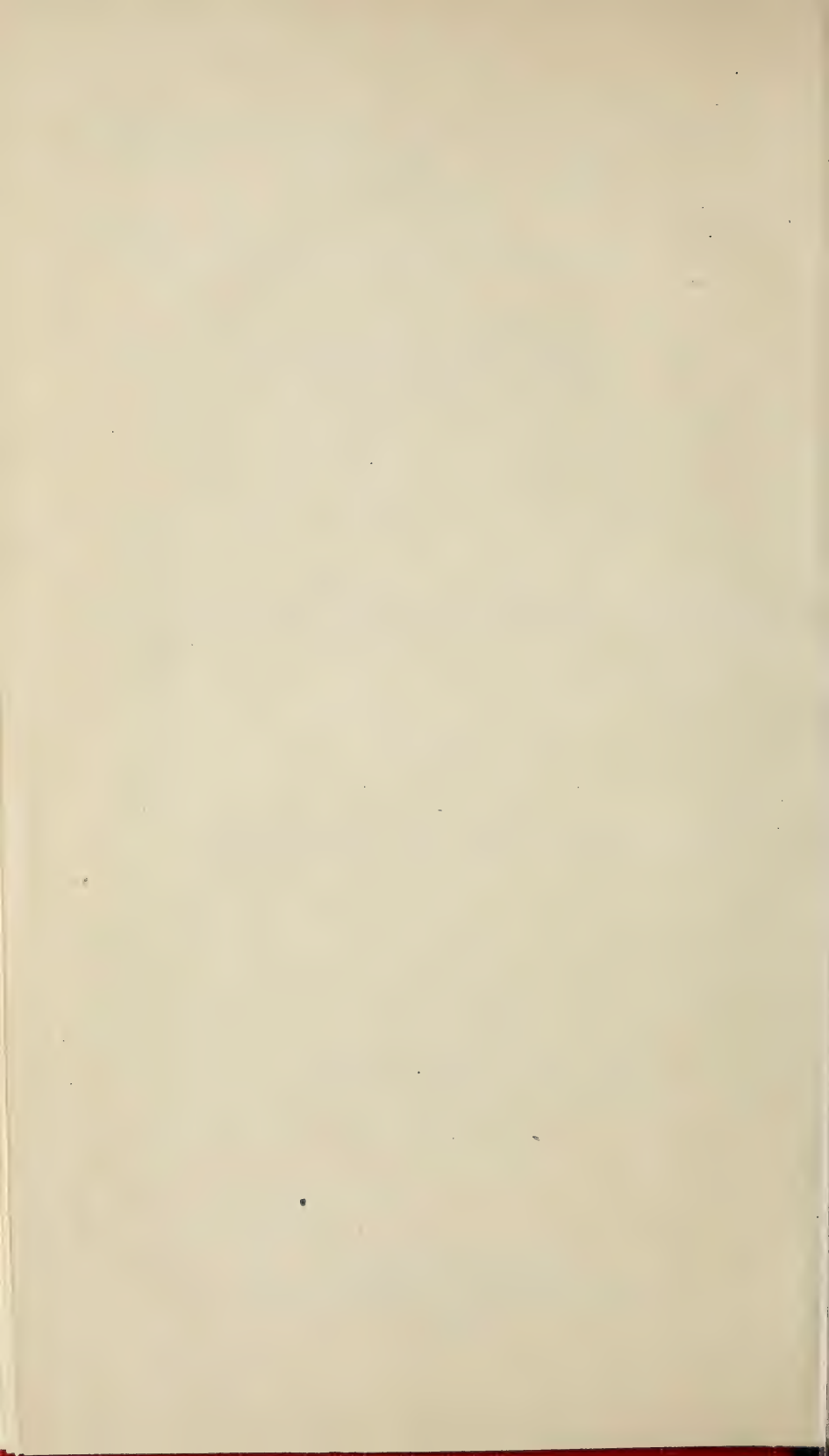
PART FIRST:

DAIRY PRODUCTS.

WASHINGTON:
GOVERNMENT PRINTING OFFICE,

1887.





LETTER OF SUBMITTAL.

SIR: I have the honor to submit herewith for your inspection and approval Bulletin No. 13, devoted chiefly to a discussion of the best methods of detecting the adulteration of foods.

The first part, which is now placed in your hands, treats of dairy products. Much interest has lately been manifested among those engaged in agriculture in respect of the adulteration of butter, and this part of the subject has been treated with greater detail than any other.

It has been my object in this work to determine the best methods of analysis of the various products in question, and all the recent improvements in analytical methods have been thoroughly tried, and those which have given good results have been adopted in the analytical work which has been done.

Within the last year my division has been supplied with apparatus for photo-micrography, and the illustrations in the following pages are entirely the work of the division unless otherwise stated.

Great benefit has been derived from this method of fixing photographic appearances, as the illustration of the crystalline characters of butters and butter substitutes sufficiently show. The examination of condiments, &c., the report of which will soon follow, was made almost entirely with the microscope, and the illustrations will show how satisfactory this kind of work proves to be. In the matter of photographic illustration no attempt has been made to confine the exhibition to phenomenally fine specimens, but the ordinary appearance of the field of vision has been reproduced. This, I think, is of greater advantage to the general investigation than would be the publication only of the strikingly good negatives. It is believed that by following the methods of analysis recommended in the report it will be possible to detect without fail any adulteration of butter that could possibly prove a commercial success. All other forms of adulteration will be suppressed by the laws of trade. In addition to the report herewith submitted the following parts of the bulletin are almost ready for the press, viz, condiments, sugar, sirap and honey, drinks and canned goods, flour and meal, tea and coffee, and baking powders. Other parts will follow as soon as time is afforded to submit all the process involved to a thorough examination in the laboratory.

Respectfully,

H. W. WILEY,
Chemist.

Hon. NORMAN J. COLMAN,
Commissioner of Agriculture.

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BUTTER AND ITS ADULTERATIONS.

The adulteration of butter with other fats has of late years attracted the attention not only of the analyst but also of the political economist and health officer.

This matter has been deemed of sufficient importance to demand regulation by law of Congress. This law provides for the inspection and analysis of commercial butters and their substitutes.

Following is the text of the act:

AN ACT defining butter, also imposing a tax upon and regulating the manufacture, sale, importation, and exportation of oleomargarine.

Be it enacted by the Senate and House of Representatives of the United States of America in Congress assembled, That for the purposes of this act the word "butter" shall be understood to mean the food product usually known as butter, and which is made exclusively from milk or cream, or both, with or without common salt, and with or without additional coloring matter.

SEC. 2. That for the purposes of this act certain manufactured substances, certain extracts, and certain mixtures and compounds, including such mixtures and compounds with butter, shall be known and designated as "oleomargarine," namely: All substances heretofore known as oleomargarine, oleo, oleomargarine-oil, butterine, lardine, suine, and neutral; all mixtures and compounds of oleomargarine, oleo, oleomargarine-oil, butterine, lardine, suine, and neutral; all lard extracts and tallow extracts; and all mixtures and compounds of tallow, beef-fat, suet, lard, lard-oil, vegetable-oil, annatto, and other coloring matter, intestinal fat, and offal fat made in imitation or semblance of butter, or when so made, calculated, or intended to be sold as butter or for butter.

SEC. 3. That special taxes are imposed as follows:

Manufacturers of oleomargarine shall pay six hundred dollars. Every person who manufactures oleomargarine for sale shall be deemed a manufacturer of oleomargarine.

Wholesale dealers in oleomargarine shall pay four hundred and eighty dollars. Every person who sells or offers for sale oleomargarine in the original manufacturer's packages shall be deemed a wholesale dealer in oleomargarine. But any manufacturer of oleomargarine who has given the required bond and paid the required special tax, and who sells only oleomargarine of his own production, at the place of manufacture, in the original packages to which the tax-paid stamps are affixed, shall not be required to pay the special tax of a wholesale dealer in oleomargarine on account of such sales.

Retail dealers in oleomargarine shall pay forty-eight dollars. Every person who sells oleomargarine in less quantities than ten pounds at one time shall be regarded as a retail dealer in oleomargarine; and sections thirty-two hundred and thirty-two, thirty-two hundred and thirty-three, thirty-two hundred and thirty-four, thirty-two hundred and thirty-five, thirty-two hundred and thirty-six, thirty-two hundred and thirty-seven, thirty-two hundred and thirty-eight, thirty-two hundred and thirty-

nine, thirty-two hundred and forty, thirty-two hundred and forty-one, and thirty-two hundred and forty-three of the Revised Statutes of the United States are, so far as applicable, made to extend to and include and apply to the special taxes imposed by this section, and to the persons upon whom they are imposed: *Provided*, That in case any manufacturer of oleomargarine commences business subsequent to the thirtieth day of June in any year, the special tax shall be reckoned from the first day of July in that year, and shall be five hundred dollars.

SEC. 4. That every person who carries on the business of a manufacturer of oleomargarine without having paid the special tax therefor, as required by law, shall, besides being liable to the payment of the tax, be fined not less than one thousand and not more than five thousand dollars; and every person who carries on the business of a wholesale dealer in oleomargarine without having paid the special tax therefor, as required by law, shall, besides being liable to the payment of the tax, be fined not less than five hundred nor more than two thousand dollars; and every person who carries on the business of a retail dealer in oleomargarine without having paid the special tax therefor, as required by law, shall, besides being liable to the payment of the tax, be fined not less than fifty nor more than five hundred dollars for each and every offense.

SEC. 5. That every manufacturer of oleomargarine shall file with the collector of internal revenue of the district in which his manufactory is located such notices, inventories, and bonds, shall keep such books and render such returns of material and products, shall put up such signs and affix such number to his factory, and conduct his business under such surveillance of officers and agents as the Commissioner of Internal Revenue, with the approval of the Secretary of the Treasury, may, by regulation, require. But the bond required of such manufacturer shall be with sureties satisfactory to the collector of internal revenue, and in a penal sum of not less than five thousand dollars; and the sum of said bond may be increased from time to time, and additional sureties required at the discretion of the collector, or under instructions of the Commissioner of Internal Revenue.

SEC. 6. That all oleomargarine shall be packed by the manufacturer thereof in firkins, tubs, or other wooden packages not before used for that purpose, each containing not less than ten pounds, and marked, stamped, and branded as the Commissioner of Internal Revenue, with the approval of the Secretary of the Treasury, shall prescribe; and all sales made by the manufacturers of oleomargarine, and wholesale dealers in oleomargarine, shall be in original stamped packages. Retail dealers in oleomargarine must sell only from original stamped packages, in quantities not exceeding ten pounds, and shall pack the oleomargarine sold by them in suitable wooden or paper packages, which shall be marked and branded as the Commissioner of Internal Revenue, with the approval of the Secretary of the Treasury, shall prescribe. Every person who knowingly sells or offers for sale, or delivers or offers to deliver, any oleomargarine in any other form than in new wooden or paper packages as above described, or who packs in any package any oleomargarine in any manner contrary to law, or who falsely brands any package or affixes a stamp on any package denoting a less amount of tax than that required by law, shall be fined for each offense not more than one thousand dollars, and be imprisoned not more than two years.

SEC. 7. That every manufacturer of oleomargarine shall securely affix, by pasting, on each package containing oleomargarine manufactured by him, a label on which shall be printed, besides the number of the manufactory and the district and State in which it is situated, these words: "Notice—The manufacturer of the oleomargarine herein contained has complied with all the requirements of law. Every person is cautioned not to use either this package or the stamp thereon again, nor to remove the contents of this package without destroying said stamp, under the penalty provided by law in such cases." Every manufacturer of oleomargarine who neglects to affix such label to any package containing oleomargarine made by him, or sold or offered for sale by or for him, and every person who removes any such label so affixed

from any such package, shall be fined fifty dollars for each package in respect to which such offense is committed.

SEC. 8. That upon oleomargarine which shall be manufactured and sold, or removed for consumption or use, there shall be assessed and collected a tax of two cents per pound, to be paid by the manufacturer thereof; and any fractional part of a pound in a package shall be taxed as a pound. The tax levied by this section shall be represented by coupon stamps; and the provisions of existing laws governing the engraving, issue, sale, accountability, effacement, and destruction of stamps relating to tobacco and snuff, as far as applicable, are hereby made to apply to stamps provided for by this section.

SEC. 9. That whenever any manufacturer of oleomargarine sells, or removes for sale or consumption, any oleomargarine upon which the tax is required to be paid by stamps, without the use of the proper stamps, it shall be the duty of the Commissioner of Internal Revenue, within a period of not more than two years after such sale or removal, upon satisfactory proof, to estimate the amount of tax which has been omitted to be paid, and to make an assessment therefor and certify the same to the collector. The tax so assessed shall be in addition to the penalties imposed by law for such sale or removal.

SEC. 10. That all oleomargarine imported from foreign countries shall, in addition to any import duty imposed on the same, pay an internal-revenue tax of fifteen cents per pound, such tax to be represented by coupon stamps as in the case of oleomargarine manufactured in the United States. The stamps shall be affixed and canceled by the owner or importer of the oleomargarine while it is in the custody of the proper custom-house officers; and the oleomargarine shall not pass out of the custody of said officers until the stamps have been so affixed and canceled, but shall be put up in wooden packages, each containing not less than ten pounds, as prescribed in this act for oleomargarine manufactured in the United States, before the stamps are affixed; and the owner or importer of such oleomargarine shall be liable to all the penal provisions of this act prescribed for manufacturers of oleomargarine manufactured in the United States. Whenever it is necessary to take any oleomargarine so imported to any place other than the public stores of the United States for the purpose of affixing and canceling such stamps, the collector of customs of the port where such oleomargarine is entered shall designate a bonded warehouse to which it shall be taken, under the control of such customs officer as such collector may direct; and every officer of customs who permits any such oleomargarine to pass out of his custody or control without compliance by the owner or importer thereof with the provisions of this section relating thereto, shall be guilty of a misdemeanor, and shall be fined not less than one thousand dollars nor more than five thousand dollars, and imprisoned not less than six months nor more than three years. Every person who sells or offers for sale any imported oleomargarine, or oleomargarine purporting or claimed to have been imported, not put up in packages and stamped as provided by this act, shall be fined not less than five hundred dollars nor more than five thousand dollars, and be imprisoned not less than six months nor more than two years.

SEC. 11. That every person who knowingly purchases or receives for sale any oleomargarine which has not been branded or stamped according to law shall be liable to a penalty of fifty dollars for each such offense.

SEC. 12. That every person who knowingly purchases or receives for sale any oleomargarine from any manufacturer who has not paid the special tax shall be liable for each offense to a penalty of one hundred dollars, and to a forfeiture of all articles so purchased or received, or of the full value thereof.

SEC. 13. That whenever any stamped package containing oleomargarine is emptied, it shall be the duty of the person in whose hands the same is to destroy utterly the stamps thereon; and any person who willfully neglects or refuses so to do shall for each such offense be fined not exceeding fifty dollars, and imprisoned not less than ten days nor more than six months. And any person who fraudulently gives away or accepts

from another, or who sells, buys, or uses for packing oleomargarine, any such stamped package, shall for each such offense be fined not exceeding one hundred dollars, and be imprisoned not more than one year. Any revenue officer may destroy any emptied oleomargarine package upon which the tax-paid stamp is found.

SEC. 14. That there shall be in the office of the Commissioner of Internal Revenue an analytical chemist and a microscopist, who shall each be appointed by the Secretary of the Treasury, and shall each receive a salary of two thousand five hundred dollars per annum; and the Commissioner of Internal Revenue may, whenever in his judgment the necessities of the service so require, employ chemists and microscopists, to be paid such compensation as he may deem proper, not exceeding in the aggregate any appropriation made for that purpose. And such Commissioner is authorized to decide what substances, extracts, mixtures, or compounds which may be submitted for his inspection in contested cases are to be taxed under this act; and his decision in matters of taxation under this act shall be final. The Commissioner may also decide whether any substance made in imitation or semblance of butter, and intended for human consumption, contains ingredients deleterious to the public health; but in case of doubt or contest his decisions in this class of cases may be appealed from to a board hereby constituted for the purpose, and composed of the Surgeon-General of the Army, the Surgeon-General of the Navy, and the Commissioner of Agriculture, and the decisions of this board shall be final in the premises.

SEC. 15. That all packages of oleomargarine subject to tax under this act that shall be found without stamps or marks as herein provided, and all oleomargarine intended for human consumption which contains ingredients adjudged, as hereinbefore provided, to be deleterious to the public health, shall be forfeited to the United States. Any person who shall willfully remove or deface the stamps, marks, or brands on package containing oleomargarine taxed as provided herein shall be guilty of a misdemeanor, and shall be punished by a fine of not less than one hundred dollars nor more than two thousand dollars, and by imprisonment for not less than thirty days nor more than six months.

SEC. 16. That oleomargarine may be removed from the place of manufacture for export to a foreign country without payment of tax or affixing stamps thereto, under such regulations and the filing of such bonds and other security as the Commissioner of Internal Revenue, with the approval of the Secretary of the Treasury, may prescribe. Every person who shall export oleomargarine shall brand upon every tub, firkin, or other package containing such article the word "oleomargarine," in plain Roman letters not less than one half inch square.

SEC. 17. That whenever any person engaged in carrying on the business of manufacturing oleomargarine defrauds, or attempts to defraud, the United States of the tax on the oleomargarine produced by him, or any part thereof, he shall forfeit the factory and manufacturing apparatus used by him, and all oleomargarine and all raw material for the production of oleomargarine found in the factory and on the factory premises, and shall be fined not less than five hundred dollars nor more than five thousand dollars, and be imprisoned not less than six months nor more than three years.

SEC. 18. That if any manufacturer of oleomargarine, any dealer therein or any importer or exporter thereof shall knowingly or willfully omit, neglect, or refuse to do, or cause to be done, any of the things required by law in the carrying on or conducting of his business, or shall do anything by this act prohibited, if there be no specific penalty or punishment imposed by any other section of this act for the neglecting, omitting, or refusing to do, or for the doing or causing to be done, the thing required or prohibited, he shall pay a penalty of one thousand dollars; and if the person so offending be the manufacturer of or a wholesale dealer in oleomargarine, all the oleomargarine owned by him, or in which he has any interest as owner, shall be forfeited to the United States.

SEC. 19. That all fines, penalties, and forfeitures imposed by this act may be recovered in any court of competent jurisdiction.

SEC. 20. That the Commissioner of Internal Revenue, with the approval of the Secretary of the Treasury, may make all needful regulations for the carrying into effect of this act.

SEC. 21. That this act shall go into effect on the ninetieth day after its passage; and all wooden packages containing ten or more pounds of oleomargarine found on the premises of any dealer on or after the ninetieth day succeeding the date of the passage of this act shall be deemed to be taxable under section eight of this act, and shall be taxed, and shall have affixed thereto the stamps, marks, and brands required by this act or by regulations made pursuant to this act; and for the purposes of securing the affixing of the stamps, marks, and brands required by this act, the oleomargarine shall be regarded as having been manufactured and sold, or removed from the manufactory for consumption or use, on or after the day this act takes effect; and such stock on hand at the time of the taking effect of this act may be stamped, marked, and branded under special regulations of the Commissioner of Internal Revenue, approved by the Secretary of the Treasury; and the Commissioner of Internal Revenue may authorize the holder of such packages to mark and brand the same and to affix thereto the proper tax-paid stamps.

Approved, August 2, 1886.

ARTIFICIAL BUTTER.

The French chemist, Mège-Mouries, in 1870 first described a method of making artificial butter on a large scale.

Mège, who was employed on the Imperial farm at Vincennes, was led to undertake this study through a desire to furnish to the poorer classes and to sailors an article which should be cheaper and more stable in its composition than ordinary butter.

He endeavored to imitate the physiological process which he supposed took place when cows were insufficiently fed, and when, therefore, the butter which they furnished was derived from their own fat. From beef he obtained a fat "which melted at almost the exact temperature of butter, possessed a sweet and agreeable taste, and which for most purposes could replace ordinary butter, not, of course, the finest kinds, but which was superior to it in possessing the advantageous peculiarity of keeping for a long time without becoming rancid."

Before the breaking out of the Franco-Prussian war Mège had established a factory at Poissy. The war suspended the operations of this factory, but at the cessation of hostilities they were again commenced.

Following is the method employed in the year 1873, in the manufacture of artificial butters:

The fat of best quality from recently killed bullocks is finely cut in a kind of sausage grinder in order to break up the membranes. The fragments fall into a tank heated with steam, which for every 1,000 parts of fat contains 300 parts of water and 1 part of carbonate of potash and 2 stomachs of sheep or pigs.

The temperature of the mixture is raised to 45° C. After two hours, under the influence of the pepsin in the stomachs, the membranes are dissolved and the fat melted and risen to the top of the mixture.

The fat is next drawn off into a second tank, kept at a somewhat higher temperature, and 2 per cent. of common salt added. After two

hours more the fat becomes clear and takes on a yellow color and acquires somewhat the taste and odor of fresh butter. The fat is now drawn off into vessels and allowed to cool. It is then cut into pieces, wrapped in linen, and put in a hydraulic press and kept at a temperature of about 25° C. By pressure the fat is separated into two portions, viz: stearine 40 to 50 per cent., and fluid oleo 50 to 60 per cent. The stearine remaining in the presses is used in candle-making. Mége's patent, possessing as it does historical interest, is given in full.

A full citation of the various patents taken out in foreign countries is found in "Sell's Kunstbutter."¹

The patents taken out in this country for the manufacture of artificial butter are given below:

LIST OF PATENTS GRANTED IN THE UNITED STATES FOR THE MANUFACTURE OF BUTTER SUBSTITUTES.

Hippolyte Mége, No. 146012, dated December 30, 1873.

To all whom it may concern:

Be it known that I, Hippolyte Mége, of Paris, France, have discovered a new and improved process of transforming animal fats into butter, of which the following is a full, clear, and exact description:

The butter which is obtained from milk is produced by the cow elaborating her own fat through her cellular mammary tissues at the low rate of temperature of the body.

The animal fat from which the butter-cells in milk are produced is composed chiefly of oleine, margarine, and stearine, and small quantities of other substances.

The natural process performed by the cow consists, mainly, first, in separating the oleomargarine from the stearine without developing disagreeable odors or flavors in the oleomargarine; and, secondly, in producing a slight change in the oleomargarine, by which it assumes the character of butter.

My invention, hereinafter described, is based upon a discovery made by me, that when the fat is rendered at a low temperature, considerably below that heretofore employed in the ordinary rendering of fat, it has the taste of molten butter, and does not acquire that peculiarly disagreeable flavor heretofore supposed to be necessarily attached to melted fat or tallow, and which is designated as "tallowy flavor."

I have succeeded in obtaining excellent results by rendering the crude fat at a temperature of 103° Fahrenheit, which is below the temperature at which the tallowy flavor is created. The temperature may be raised above this point in order to facilitate the operation, provided care be taken to avoid attaining the temperature at which the tallowy flavor is created.

The precise limit to which it is safe to increase the rendering-temperature can be ascertained by trial under various circumstances with the different kinds of fat. The temperature must, however, be far below that heretofore ordinarily used in rendering fats when no such object as I propose—to wit, the making of a butter-like product—was had in view. I do not think it would be safe to vary many degrees above that specifically indicated.

I have also discovered that, in order to neutralize any fermentation of the fat before or during its treatment, the raw fat should, as soon as possible after the death of the animal, be plunged in a solution of fifteen (15) per cent. of common salt and one per cent. of sulphate of soda, the effect of which would be to prevent such fermentation.

¹Arbeiten a. d. Kaiserlichen Gesundheitsamte, pp. 481-493.

In carrying out my process I first crush, grind, or disintegrate the fat by any suitable machinery, such as rollers or millstones, in order to break up the cellular tissues in which the fat is contained in the animal, and thus cause it to be more easily melted or rendered by the application of low temperatures. This fat thus disintegrated is to be slowly raised to a temperature of 103° Fahrenheit in a vessel in which the temperature can be raised at will until the rendering shall be complete. The temperature, as before stated, must be so regulated that the rendered fat will have the taste of molten butter, and care should be taken not to heat it so as to induce the change which produces the usual disagreeable taste of melted fat or tallow, instead of the taste of molten butter, which temperature is considerably below that heretofore ordinarily used in rendering fat, and will be found to vary not many degrees above the point already stated.

I also add to the fat while being rendered, for the purpose of aiding in this process, two liters of gastric juice to a hundred (100) kilograms of fat. This gastric juice is made by macerating, for three hours, half of the stomach of a pig or sheep, well washed, and three litres of water containing thirty grams of bi-phosphate of lime. After maceration this macerated substance is passed through a sieve, and then added to the fat under treatment in the proportion of two litres to one hundred (100) kilograms.

The separation of the organized tissues from the fat is aided by the introduction of salt during the rendering; and as soon as there are no lumps of fat visible in the kettle I add about one per cent. of common salt. I stir it for some time. The rendered fat is then allowed to stand until it attains perfect limpidity, when it can be drawn off. By this means the separation is well made, and the organized tissues which do deposit are not altered. I then allow the melted fat to stand in a vessel, maintained at a temperature of about 86° to 98° , until the stearine is crystallized. The mixture of stearine and oleomargarine may then be put in a centrifugal machine; and by the operation of this machine the oleomargarine will pass through the cloth and the stearine remain within; or the mixture may be subjected to pressure in a press. The effect produced in either case is that the oleomargarine practically separates from the stearine and flows out. The oleomargarine thus separated from the stearine, when cooled, constitutes a fatty matter of very good taste, which may replace the butter used in the kitchen. If it is desired, however, to transform it into more perfect butter, I employ the following means: I mix the oleomargarine, as it comes from the press or centrifugal machine, with milk and cream, equal to ten per cent. of the weight of the oleomargarine, the temperature of the milk and cream being about seventy-one (71) degrees, and thoroughly agitate them together. I then let the mixture become completely cold and solid, and then cause it to be worked between rollers, which give it the homogeneousness and the consistency which are the qualities of the natural butter.

The above process of agitating the oleomargarine with milk is intended to be adopted when the butter is to be immediately used. If the butter is intended to be preserved, it will be better to mix the oleomargarine at animal heat with ten per cent. of its weight of water instead of milk or cream, and then agitate the two together, as above described.

I have also found it expedient to mix with the cream or milk, in the first case above described, before agitating, or with the water in the other case above described, before agitating, a fiftieth part of mammary tissue, which is the udder of the cow, minced fine, a one-hundredth part of bicarbonate of soda, and some coloring matter.

It may be desirable to add ordinary butter, and this I do by mixing the oleomargarine and the ordinary butter together at a temperature of about 70° Fahrenheit.

What I claim as my invention, and desire to secure by Letters Patent, is—

- (1) The rendering of animal fat at a low temperature, substantially as above set forth, for the production of a fatty matter devoid of disagreeable taste.
- (2) As a new product of manufacture, fat rendered at the low temperature, substantially as above described, devoid of disagreeable taste.
- (3) The combined process of rendering animal fat at a low temperature and then separating the oleomargarine for the purpose of producing a material adapted to be used as ordinary butter for culinary purposes, or to be further treated for making more perfect butter, substantially as above described.
- (4) As a new product of manufacture, oleomargarine obtained from fat rendered at a low temperature and separated from the stearine, substantially as above described.
- (5) The agitating of oleomargarine with water or milk for the purpose of making a more perfect imitation of butter, substantially as above described.
- (6) The butter-like product produced by the agitation of oleomargarine with water or milk, substantially as above described.
- (7) The treatment with artificial gastric juice for facilitating the process of rendering the fat at a low temperature, substantially as above described.
- (8) The treatment of the oleomargarine with the mammary tissue of the cow, or mammary pepsin, substantially as above described.
- (9) The addition of ordinary butter to oleomargarine, substantially as above described.

H. MÉGE.

Witnesses :

ROBT. M. HOOPER,

M. D. DESHLER.

William E. Andrew, No. 153,999, dated August 11, 1874.

The process, herein described, for rendering fats, consisting in the application of dry heat or dry hot air to liquefy, and pressure to separate, the oily portion from the membrane, and removing the liquid portions from contact with the membranous portions as fast as separated.

William E. Andrew, No. 166,955, dated August 24, 1875.

Complete process of manufacturing artificial butter, herein described, consisting first in rupturing and destroying the globular condition of animal oil by agitation and then refrigerating the same, then combining the product thus obtained with butter, cream, or milk and churning until a thorough amalgamation takes place.

William E. Andrew, No. 172,942, dated February 1, 1876.

The process of clarifying liquid tallow or oil by injecting into the oil, under force, in the form of mist or fine spray, water prepared with chloride of sodium or nitrate of potash, and heated to a higher degree of temperature than the oil.

Garret Cosine, No. 173,591, dated February 15, 1876.

The process of making artificial butter by mixing together oleine and margarine from animal fats, and from fruit and vegetable nuts, and lactic acid and lopered cream or milk.

William E. Andrew, No. 179,883, dated July 18, 1876.

(Mechanical.)

Alfred Springer, No. 187,327, dated February 13, 1877.

The process of producing edible fat or tallow by heating the crude fat at a temperature of 140° to 145° Fahrenheit, in contact with common salt, saltpeter, borax, and boracic and salicylic acids, withdrawing the separated fat and incorporating therewith a second and smaller charge of the above chemicals, with the addition of benzoic acid.

Amor Smith, No. 188,428, dated March 13, 1877.

Method of separating oleomargarine from the fat of kine, that is to say, by separating it directly from the stearine and membrane at a low heat, without having first heated the mass to a higher point, for the purpose of removing the membrane from the stearine and oleine.

Royal W. Barnard, No. 198,334, dated December 18, 1877.

Method of reclaiming sour "tubby," or rancid butter, which consists in treating the same with a solution of brine containing an alkaline carbonate mixed with a solution of tartaric acid, or its equivalent.

Thomas F. Wilkins, No. 226,467, dated April 13, 1880.

Butter containing metaphosphoric acid intimately incorporated therewith, whereby the butter is preserved.

Otto Roysen, No. 236,483, dated January 11, 1881.

Process of making a substitute for butter, consisting in adding to oleomargarine an alkaline solution, and agitating the mixture until partial saponification ensues, and then adding a minute quantity of butyric acid.

Thomas F. Wilkins, No. 9,892, reissued, dated October 11, 1881.

The method herein described of preserving fats and other organic matter by mechanically mingling phosphoric acid therewith.

Samuel H. Cochran, No. 258,992, dated June 6, 1882.

The combination of beef-suet oil, cotton-seed oil and its equivalents, purified and flavored as described, with beef-stearine and slippery-elm bark.

Hippolyte Mège, No. 10,137, reissued, dated June 13, 1882.

Treating animal fats so as to remove the tissues and other portions named, with or without the addition of substances to change the flavor.

Samuel H. Cochran, No. 10,171, reissued, dated August 1, 1882.

A combination of beef-suet oil, cotton-seed oil and its equivalents, with beef-stearine.

Samuel H. Cochran, No. 262,207, dated August 8, 1882.

Compound composed of the oil obtained from swine fat, cotton-seed oil and its equivalents, deodorized and purified by slippery-elm bark and beef-stearine.

John Hobbs, No. 263,042, dated August 22, 1882.

The vegetable stearine to be used can be obtained from any pure vegetable, seed, or nut oils by pressing them at a temperature as above set forth, or it may be obtained in the market at times as vegetable stearine.

Mixing "vegetable stearine" or "margarine" obtained substantially as described, with what is called "animal oleomargarine" and emulsifying the said mixture with milk, cream or other watery fluid.

Nathan I. Nathan, No. 263,199, dated August 22, 1882.

Process of manufacturing artificial butter by uniting oleomargarine with leaf lard, the latter having been previously cleaned, fused, strained, and subjected to a washing action in a solution of water, borax, and nitric acid, then rewashd and the united mass heated and subjected to the ordinary churning operation.

George S. Marshall, No. 264,545, dated September 19, 1882.

Process of deodorizing, purifying and flavoring stearine obtained from animal fats, or vegetable oils, by boiling the same with water and mixing therewith powdered orris-root.

William Cooley, No. 264,516, dated September 19, 1882.

An artificial cream composed of an oleaginous substance mechanically blended, or otherwise incorporated with milk, buttermilk, or cream, the oleaginous material being in a state of minute and even division, and each particle encased in a coating of caseine.

Henry Lauferty, No. 265,833, dated October 10, 1882.

Improvement in the manufacture of artificial butter, or oleomargarine, which consists in treating in the manner described both the milk and the oleomargarine oil separately with sal-soda, prepared and taken in the proportions as specified, then mixing or churning the creamy substance produced from the treated milk with the prepared oleomargarine oil, and coloring, salting, and working the mixture.

Hugo Berthold, No. 266,417, dated October 24, 1882.

A coloring compound for admixture with oleomargarine oil after the usual churning operation, consisting of saccharine matter, glycerine, annatto, and oil of ben, mixed together.

George H. Webster, No. 266,568, dated October 24, 1882.

Process of making artificial butter, which consists in minutely dividing leaf-lard, rendering and straining it, mixing a butter-coloring matter with it, immersing it for thirty-six hours in cold brine, transferring it from the brine to dry tables or shelves and keeping it there covered with salt for thirty-six hours; then heating it to about 130° Fahrenheit and mixing it with lukewarm buttermilk, a small quantity of clarified tallow, and a minute quantity of pepsin, and allowing the mixture to settle; then transferring the liquid lard and tallow to a vessel containing comminuted butter of about half the weight of the lard, thoroughly mixing the contents of the vessel by stirring, pouring the mixture into cold water, and thoroughly working it in the usual manner.

William H. Burnett, No. 266,580, dated October 24, 1882.

The butter-like product described, consisting of the ingredients specified, to wit, lard, beef-suet, butter, glycerine, salt water, and coloring material.

Oscar H. Coumbe, No. 266,778, dated October 31, 1882.

A new article of manufacture, oleard, consisting of vegetable oil, in combination with cooked farinaceous flour.

Oscar H. Coumbe, No. 266,777, dated October 31, 1882.

An improved article of commerce known as butteroid, and consisting of cottonseed or other vegetable oil treated with a solution of caustic soda, in combination with farinaceous flour first thoroughly cooked in salt water.

Henry R. Wright, No. 267,637, dated November 14, 1882.

Process of making artificial butter or creamine, which consists in mixing together the oils derived from animal fat at low temperatures with sweet cream, the oil of butter, vegetable oil, and coloring matter; then allowing these ingredients to become sour while together; then removing the whey, and finally churning the mass.

Joseph H. McDonald, No. 270,454, dated January 9, 1883.

(Mechanical.)

John Hobbs, No. 271,239, dated January 30, 1883.

(Mechanical.)

John Hobbs, No. 271,240, dated January 30, 1883.

(Mechanical.)

John Hobbs, No. 271,244, dated January 30, 1883.

(Mechanical.)

John Hobbs, No. 271,241, dated January 30, 1883.

(Mechanical.)

John Hobbs, No. 271,243, dated January 30, 1883.

(Mechanical.)

John Hobbs, No. 271,242, dated January 30, 1883.

(Mechanical.)

John Hobbs, No. 280,822, dated July 10, 1883.

Process of refining fats, which consists in first finely grinding the fat of the leaf of the hog, mixing it thoroughly with salt, placing it in tanks of cold water for two or three days, when it is worked over, as described, then rendering it at a low temperature, and as quickly as possible, with or without adding the solution mentioned, then drawing it off from the tissue, clarifying it and again drawing it off and cooling it.

Samuel H. Cochran, No. 285,878, dated October 2, 1883.

The mode above described of giving a butter-flavor to animal fats or oils, which consists in mixing therewith in the manner above described a quantity of dairy or creamery butter in its normal or hard condition.

Samuel H. Cochran, No. 285,973, dated October 2, 1883.

(Mechanical.)

Samuel H. Cochran, No. 285,974, dated October 2, 1883.

(Mechanical.)

Andrew J. Chase, No. 286,778, dated October 16, 1883.

The method herein described of manufacturing butter from animal oils, said method consisting in subjecting the oils to a low temperature, and at the same time agitating them, both during the process of solidifying and afterwards.

John Hobbs, No. 289,100, dated November 27, 1883.

The manufacture of deodorized fats or oxyline, the use or employment of the substance herein mentioned—vegetable stearine—in combination with the other ingredients named—oleomargarine-stearine and oleomargarine-stock.

George Lawrence, No. 295,180, dated March 18, 1884.

Process of treating milk with fatty and other matters by passing it and them, mingled with gases, through one or more steam-ejectors, for separating and mixing the particles.

Samuel Schwarzschild, No. 299,685, dated June 3, 1884.

(Mechanical.)

Emma J. Woodruff, No. 327,636, dated October 6, 1885.

Adding to the milk white-wine rennet, sugar, salt, bicarbonate of soda, bicarbonate potassium, alum, and butter.

Lyman Guinnip, No. 334,430, dated January 19, 1886.

Consisting in mingling two bodies of cream of different age, then churning the same, then removing a portion thereof from the churn and mingling with the removed part a quantity of butter, then churning the residue until butter begins to separate, then adding butter thereto, as specified, and churning the mixture, and finally adding thereto the portion first abstracted, and churning the whole until the butter is made.

William A. Murray, No. 335,084, dated January 26, 1886.

Mixing 1 gallon of sweet milk with 1 ounce of liquid rennet, 25 grains (Troy) of nitrate of potash, 1 ounce granulated sugar, half-teaspoonful of butter-coloring, and 8 pounds of butter, churned together and worked.

Carl August Johansson, No. 336,324, dated February 16, 1886.

(Mechanical.)

George Wm. Sample, No. 336,438, dated February 16, 1886.

(Mechanical.)

Charles Marchand, No. 338,538, dated March 23, 1886.

(Mechanical.)

Edward J. Oatman, No. 346,062, dated July 20, 1886.

Producing an emulsion from milk or its derivatives and a suitable oleaginous material, which consists in thoroughly dividing and commingling the ingredients by injecting a steam jet into the mixture.

The common method of manufacture employed in this country is set forth by Armsby:¹

Although numerous patents have been taken out for the manufacture of imitation butter, and a great variety of materials have been named in the specifications, the process as now conducted is comparatively simple. The raw materials are beef-tallow, leaf-lard, and the best quality of butter, together with small amounts of milk or cream and of butter-color.

From the beef-tallow is prepared the oleomargarine oil of Mège. The caul fat of freshly killed bees is, after thorough washing first in tepid and then in iced water, allowed to stand in a cold room until thoroughly cold. It is then rendered at a temperature between 130° and 175° F. The resulting oil is allowed to cool slowly until a considerable portion of the stearine and palmitin have crystallized out, and the pasty mass is then subjected to hydraulic pressure. The still fluid (about two-thirds of the whole) flows out into a tank of cold water, where it solidifies into a granular mass which is known in the trade as "oleo" oil or simply "oleo". The name "oil" is somewhat misleading, as the product is a granular solid of a slightly yellow color. Fresh leaf-lard treated in substantially the same way as the beef-tallow, yields the "neutral lard" or "neutral" of the trade, also a granular solid of a white color.

The objects of this treatment are twofold: first, to produce fats as free as possible from taste or odor; second, to remove some of the difficultly fusible stearine and palmitin, in order that the finished product may melt readily in the mouth.

Having thus secured the fats in proper condition, the manufacturer proceeds to mix the "oleo" and "neutral", the proportions varying according to the destination of product; a warm climate calling for more "oleo," a cold one for more "neutral," and to flavor the mixture with butter. This flavoring is conducted in large, steam-jacketed vessels provided with revolving paddles, by which their contents can be thoroughly agitated. Here the "oleo" and "neutral" are melted and thoroughly agitated with a certain proportion of milk, or sometimes of cream, and a proper amount of butter-color. Forty-eight gallons of milk per 2,000 pounds of product are stated to be a common proportion. After sufficient agitation, the melted mass is run into cold water, and as it cools is broken up by paddles so as to granulate the mass. After thorough washing, it is salted and worked exactly like butter. The product is known as oleomargarine. Although it contains hardly more than a trace of butter fat, the latter flavors the whole mass so strongly that, when well salted, as it usually is, it might readily pass with an inexperienced or careless consumer for a rather flavorless butter. Oleomargarine is the cheapest product made. By adding to the material in the agitator or "churn" more or less pure butter, what is known as butterine is produced, two grades of which are commonly sold, viz, "creamery butterine" containing more, and "dairy butterine" containing less butter.

The method of manufacture used by the firm of Armour & Co., of Chicago, is thus described by Mr. Philip D. Armour:²

The fat is taken from the cattle in the process of slaughtering, and after thorough washing is placed in a bath of clean, cold water, and surrounded with ice, where it is allowed to remain until all animal heat has been removed. It is then cut into small pieces by machinery and cooked at a temperature of about 150° until the fat, in liquid form, has separated from the fibrine or tissue, then settled until it is perfectly clear. Then it is drawn into graining vats and allowed to stand a day, when it is ready for the presses. The pressing extracts the stearine, leaving the remaining product, which is commercially known as oleo oil, which, when churned with cream or milk or both and with usually a proportion of creamery butter, the whole being properly salted, gives the new food-product, oleomargarine.

¹ Science, vol. 7, pp. 471-472.

² Senate Mis. Doc. No. 131, Forty-ninth Congress, first session, p. 224.

In making butterine we use neutral lard, which is made from selected leaf lard in a very similar manner to oleo oil, excepting that no stearine is extracted. This neutral lard is cured in salt brine for forty-eight to seventy hours at an ice-water temperature. It is then taken, and, with the desired proportion of oleo oil and fine butter, is churned with cream and milk, producing an article which, when properly salted and packed, is ready for market.

In both cases coloring matter is used, which is the same as that used by dairymen to color their butter. At certain seasons of the year, viz, in cold weather, a small quantity of salad oil made from cotton seed is used to soften the texture of the product, but this is not generally used by us.

Gustavus F. Swift, of the firm of Swift & Co., of the town of Lake (near Chicago), describes as follows the method in use in the manufacture of artificial butter by his company:¹

The fat is taken from the cattle in the process of slaughtering, and after thorough washing is placed in a bath of clean, cold water and surrounded with ice, where it is allowed to remain until all animal heat has been removed. It is then cut into small pieces by machinery and cooked at a temperature of about 150° until the fat in liquid form has separated from the fibrine or tissue; then settled until it is perfectly clear. Then it is drawn into draining vats and allowed to stand a day, when it is ready for the presses. The pressing extracts the stearine, leaving the remaining product, which is commercially known as oleo oil, which, when churned with cream or milk, or both, and with usually a proportion of creamery butter, the whole being properly salted, gives the new food product, oleomargarine.

In making butterine we use neutral lard, which is made from selected leaf-lard in a very similar manner to oleo oil, excepting that no stearine is extracted. This neutral lard is cured in salt brine for forty-eight to seventy hours at an ice-water temperature. It is then taken, and with the desired proportion of oleo oil and fine butter, is churned with cream and milk, producing an article which, when properly salted and packed, is ready for market.

In both cases coloring matter is used, which is the same as that used by dairymen to color their butter. At certain seasons of the year, viz, in cold weather, a small quantity of sesame oil, or salad oil made from cotton seed, is used to soften the texture of the product.

WHOLESOMENESS OF ARTIFICIAL BUTTER.

On this subject there is a wide difference of opinion. It is undoubtedly true that a great deal of artificial butter has been thrown upon the market that has been carelessly made, and therefore harmful to the health. On the other hand a butter substitute, made carefully out of the fat of a perfectly healthy bullock or swine, is not prejudicial to health.

Prof. Henry Morton, of the Stevens Institute, Hoboken, N. J., made the following statements before the Senate Committee on Agriculture, pending the consideration of the "Oleomargarine" bill:²

The subject is one which has been of great interest to all scientific men from the time of the original discovery by Mège, which was made, as you are aware, during the siege of Paris. Many persons have been interested in it and have followed it up. I have been frequently called upon to examine processes and superintend operations where modifications in the manufacture have been suggested, and so on, and specimens have been brought to me as a chemist, to examine from time to time microscopic-

¹ *Op. cit.*, p. 225.

² *Op. cit.*, p. 47.

ally and chemically. When the substance was first introduced, the question was raised as to whether it could be distinguished from butter by any test, and I was led in that way to investigate the subject, and to examine as to all the properties which it exhibited, as well as to compare different samples of it, and I have in my experiments in this line examined great numbers of specimens of oleomargarine prepared as butter and of oleomargarine oil for the preparation of butter, from all parts of the country, and also have visited factories very frequently and spent long periods there. I have remained as long as a week in one of these factories continuously—sometimes spending the night as well as the day there, in order to watch the process completely and see the operation from beginning to end, to see what was put in and what was not, and to observe what was done and what was not done.

In the course of these examinations I have reached the conclusion, founded on these observations, that the material is of necessity, a pure one, and cannot possibly be unwholesome, and is, in fact, in that sense a thoroughly desirable and safe article of food. I will express as briefly as I can my reasons for this opinion, and state the facts on which they are founded.

In the first place I have found, as a matter of observation, that fat which is to be used in the manufacture of oleomargarine, if it is in the slightest degree tainted before the manufacture begins, if it is not strictly fresh, if it is not taken almost directly from the slaughtered animal, if it is allowed to stand in a barrel for a few hours in ordinary weather or in cold weather, if put in a barrel with any animal heat in it for a few hours, then an incipient change begins which, in the succeeding process, is exaggerated so that an utterly offensive material is produced, which could not be used for any such purpose.

Prof. C. F. Chandler says: ¹

In all of these reports I have taken the ground that this is a new process for making an old article, and that article is butter. This is a new process for making butter. It is made of materials which are in every respect wholesome and proper articles of food, whether it be made solely from the oleomargarine extracted from beef fat, or whether it has added to it more or less leaf lard properly prepared, or more or less sesame oil or cotton-seed oil, and whether it be or not colored with annatto or the other coloring matters used. I take the ground that there is nothing in any one of these materials in any sense unwholesome, and nothing in any one of them which makes it inferior as an article of food to dairy butter. I regard the discovery of Mège-Mouries, of a process by which beef fat and hog fat can be extracted from a dipose tissue and converted into a wholesome article of food free from any disagreeable taste or odor, as one of the most important discoveries made in this century, a discovery by which it is possible to make a perfectly pure and satisfactory, as well as wholesome, article of food at a reasonable price. I have visited various factories where this article is manufactured, from the time the industry began down to date. I am perfectly familiar with the materials employed and the different processes, and know there is nothing whatever used either in material or process which is unwholesome or in any way deleterious to the public health.

Professor Chandler further has reported as follows to the Board of Health of New York City: ²

NEW YORK, May 2, 1881.

To the Board of Health of the Health Department :

Having been directed by this board to investigate the subject of oleomargarine, in response to the resolutions of the Board of Aldermen, I would beg leave to submit the following report :

The resolutions directing the inquiry are as follows:

Whereas there is existing at the present time in the minds of the public great alarm and distrust in relation to the adulteration of food products ; and

¹ *Op. cit.*, p. 67.

² *Op. cit.*, p. 70.

Whereas the committee on public health of the assembly of this State has been for some time investigating the adulteration of food products, and especially oleomargarine; and

Whereas this committee have conducted such investigation by calling as witnesses principally dealers in butter and have not examined as witnesses medical or chemical experts to determine the value of oleomargarine as food; therefore

Resolved, That the board of health of this city be, and they are hereby requested and directed to take immediate measures to investigate in the most thorough manner, by medical and chemical aid, the purity, healthfulness, and value of said product as an article of food, and to report to this body the results of their investigation, with such recommendations, if any be necessary, as may relate to the manufacture and distribution of the same as an article of food.

This subject has been before the board on former occasions, and I have little to add to what has been previously stated.

Oleomargarine, invented by the distinguished French chemist, Mège-Mouries, is manufactured in New York City in a few large establishments. The material is fresh beef suet, brought directly from the slaughter-houses. It is thoroughly washed, rendered very carefully, strained to remove a portion of the hard stearine, and then churned with milk to convert it into artificial butter, which contains the same constituents as dairy butter. The process is extremely ingenious and simple and executed by machinery. Nothing objectionable exists in the original material, nor is anything objectionable added during the process, and the operations are conducted with the utmost cleanliness. The product is palatable and wholesome, can be made of uniform quality the year round, is in every respect superior as an article of food to a large proportion of dairy butter sold in this city, and can be manufactured at a much lower price. I regard it as a most valuable article of food and consider it entirely unexceptional in every respect. In this opinion I am supported by the best scientific authorities in the country. The following distinguished chemists, after carefully studying the manufacture, have made the most decided statements in favor of this new article of food:

Prof. George F. Barker, University of Pennsylvania.

Dr. Henry A. Mott, jr., New York.

Prof. G. C. Caldwell, Cornell University, Ithaca, N. Y.

Prof. S. W. Johnson, Yale College, New Haven, Conn.

Prof. C. A. Goessmann, Massachusetts Agricultural College, Amherst, Mass.

Prof. Henry Morton, Stevens Institute, Hoboken, N. J.

Prof. Charles P. Williams, Philadelphia, Pa.

Prof. W. O. Atwater, Wesleyan University, Middletown, Conn.

Prof. J. W. S. Arnold, University of New York.

I would further say that this question is one on which there is no difference of opinion among scientific investigators familiar with the chemistry of dairy products and fats. I have never seen a statement emanating from any person having any standing among scientific men in which a contrary opinion is advanced. There has recently been a very strong confirmation of my opinion published in England. A bill came before the House of Commons in England, directed against this kind of butter from America, and, after considerable discussion, was defeated by a vote of 75 to 59. In the discussion the strongest opponent to legislation against it was Dr. Lyon Playfair, one of the most distinguished chemists and sanitary authorities in England. A pupil of Graham and Leibig, he has filled the chairs of chemistry in the Royal Institution of Manchester and at the University of Edinburgh, was appointed chemist to the Museum of Practical Geology by Sir Robert Peel, represented the universities of Edinburgh and Aberdeen in Parliament, was postmaster-general in the first Gladstone cabinet, has been member of several sanitary commissions, and is now a leading member of Parliament. In his remarks he stated that "bad butter was a fraud upon the poor, and oleomargarine would sooner or later drive it out of the

market;" he "thought that good oleomargarine at one shilling a pound was a great deal better and cheaper than bad butter at one shilling four pence a pound," and he said that "as a general rule the former (oleomargarine) did not become so readily rancid as the latter (butter)."

I would further state that, as there is nothing unwholesome in oleomargarine, no legislation in regard to this article is necessary to protect the public health.

C. F. CHANDLER,
President.

Prof. G. F. Barker says:¹

UNIVERSITY OF PENNSYLVANIA,
Philadelphia, March 22, 1880.

To the United States Dairy Company:

GENTLEMEN: In reply to your inquiry, I would say that I have been acquainted for several years with the discovery of Mège-Mouries for producing butterine from oleomargarine fat. In theory the process should yield a product resembling butter in all essential respects, having identically the same fatty constituents. The butterine prepared under the inventor's patents is, therefore, in my opinion, quite as valuable a nutritive agent as butter itself. In practice the process of manufacture, as I have witnessed it, is conducted with care and great cleanliness. The butterine produced is pure and of excellent quality, is perfectly wholesome, and is desirable as an article of food. I can see no reason why butterine should not be an entirely satisfactory equivalent for ordinary butter, whether considered from the physiological or commercial standpoint.

Prof. G. C. Caldwell, of Cornell University, gives the following testimony:²

CHEMICAL LABORATORY, CORNELL UNIVERSITY,
Ithaca, N. Y., March 20, 1880.

I have witnessed, in all its stages, the manufacture of "oleomargarine" and of oleomargarine butter or "butterine."

The process for oleomargarine, when properly conducted, as in the works of the Commercial Manufacturing Company, is cleanly throughout, and includes every reasonable precaution necessary to secure a product entirely free from animal tissue, or any other impurity, and which shall consist of pure fat made up of the fats commonly known as oleine and margarine. It is, when thus prepared, a tasteless and inodorous substance, possessing no qualities whatever that can make it in the least degree unwholesome when used in reasonable quantities as an article of food.

In the manufacture of butterine, since nothing but milk, annatto, and salt, together with, perhaps, a little water from clean ice, are added to this oleomargarine, to be intimately mixed with it by churning and other operations, I have no hesitation in affirming that this also, when properly made according to the Mège patent, and other patents held by the United States Dairy Company, and when used in reasonable quantities, is a perfectly wholesome article of food; and that, while not equal to fine butter in respect to flavor, it nevertheless contains all the essential ingredients of butter, and since it contains a smaller proportion of volatile fats than is found in genuine butter it is, in my opinion, less liable to become rancid.

It cannot enter into competition with fine butter; but in so far as it may serve to drive poor butter out of the market, its manufacture will be a public benefit.

Prof. S. W. Johnson, of Yale College, makes the following statement:³

SIEFFIELD SCIENTIFIC SCHOOL OF YALE COLLEGE,
New Haven, Conn., March 20, 1880.

The United States Dairy Company:

GENTLEMEN: I am acquainted with the process discovered by M. Mège for producing the article known in commerce as oleomargarine or butterine.

¹ *Op. cit.*, p. 73.

² *Op. cit.*, p. 73.

³ *Op. cit.*, p. 74.

I have witnessed the manufacture in all its stages, as carried out on the large scale, and I can assert that when it is conducted according to the specifications of M. Mège it cannot fail to yield a product that is entirely attractive and wholesome as food, and one that is for all ordinary culinary and nutritive purposes the full equivalent of good butter made from cream.

Oleomargarine butter has the closest resemblance to butter made from cream in the external qualities—color, flavor, and texture. It has the same appearance under the microscope, and in chemical composition differs not in the nature, but only in the proportions of its components. It is, therefore, fair to pronounce them essentially identical.

While oleomargarine contains less of those flavoring principles which characterize the choicest butter, it is, perhaps for that very reason, comparatively free from the tendency to change and taint, which speedily renders a large proportion of butter unfit for human food.

I regard the manufacture of oleomargarine or butterine as a legitimate and beneficent industry.

S. W. JOHNSON,
*Professor of Theoretical and Agricultural Chemistry,
Director of the Connecticut Agricultural Experiment Station.*

Dr. C. A. Goessmann, of Amherst, indorses in general the above statements:¹

AMHERST, MASS., March 20, 1880.

United States Dairy Company, New York:

GENTLEMEN: I have visited on the 17th and 18th of the present month your factory, on West Forty-eighth street, for the purpose of studying your mode of applying Mège's discovery for the manufacture of oleomargarine butter or butterine. A careful examination into the character of the material turned to account, as well as into the details of the entire management of the manufacturing operation, has convinced me that your product is made with care, and furnishes thus a wholesome article of food. Your oleomargarine butter or butterine compares in general appearance and in taste very favorably with the average quality of the better kinds of the dairy butter in our markets. In its composition it resembles that of the ordinary dairy butter; and in its keeping quality, under corresponding circumstances, I believe it will surpass the former, for it contains a smaller percentage of those constituents (glycerides of volatile acids) which, in the main, cause the well-known rancid taste and odor of a stored butter.

I am, very respectfully, yours,

C. A. GOESSMANN, PH. D.,
Professor of Chemistry.

To these I may add the names of Prof. Charles P. Williams, of the State University of Missouri, Dr. Henry Mott, jr., Prof. W. O. Atwater, and Prof. J. W. S. Arnold.²

Armsby³ says in respect of the healthfulness of oleomargarine:

Very exaggerated and absurd statements have been made regarding the unhealthfulness of butterine and oleomargarine. The charges have in general been that the fat used is practically uncooked, and that raw animal fat is unwholesome; that filthy fat and fat from diseased animals are used, and that the product contains, or is liable to contain, the germs of disease; and that in cleansing these diseased and filthy fats dangerous chemicals are used, which are not subsequently completely removed.

That the fats used are of themselves unwholesome there is no proof whatever. They contain nothing that butter-fat does not also contain, and differ from it only by the absence of about 6 per cent. of the glyceride of certain soluble fatty acids, viz, capric, myristic, and lauric.

¹ *Op. cit.*, p. 74.

² *Op. cit.*, pp. 73, 74, 75.

³ *Science*, vol. 7, No. 172.

caprylic, capronic, and butyric acids. The only experiments upon the digestibility of imitation butter are two, by A. A. Mayer, upon oleomargarine. These showed a difference of only about 2 per cent. in favor of butter. That the higher flavor of butter acting upon the nervous system would give it a greater nutritive value than the flavorless "neutral" or "oleo" may be conceded; but that an article which even experts fail to distinguish from genuine butter is at any serious disadvantage in this respect may well be doubted.

The manufacturers claim that imitation butter can only be made from the best quality of fat from freshly-killed animals, and I know of no evidence which disproves their assertions. The sensational article recently published in a prominent agricultural paper in the Northwest, accompanied by cuts of the numerous organisms found in butterine, is of no significance in this connection, both because the species described are all harmless, and because no comparative examinations of genuine butter were made. It is highly probable that many samples of the latter would show as miscellaneous an assortment of formidable looking, harmless organisms as did the butterine.

On the other hand, however, there is at present no guaranty, except the statement of the manufacturers, that diseased fat is not or cannot be used, the manufacture being conducted entirely without any official inspection, and visitors being in most (not all) cases excluded. I believe that the chances of disease being conveyed in this way are small, but they are not yet proved to be non-existent.

As regards filthy processes of manufacture, it may safely be asserted that butterine could not successfully imitate butter were it not as clean as most things are which pass for clean in this dirty world.

The charge that dangerous chemicals are used in the manufacture may be disposed of in a few words. If a dangerous amount of any chemical which is claimed to be used were left in the finished product the latter would be inedible. Should trace of these chemicals be found their significance would not lie in themselves, but in the indication they would furnish that the original fats were impure and required chemical treatment.

Sell¹ has made an examination of the evidence for and against the unwholesomeness of artificial butter and has reached the following conclusions:

The artificial butter prepared from the fat of healthy animals, apart from possibly a somewhat less digestibility, in comparison with milk-butter furnishes in general no reason for the supposition that it can affect injuriously human health.

There is ground for the suspicion that a part of the artificial butter occurring in commerce is manufactured out of such material or by such processes as do not with certainty exclude the danger of conveying to man disease whether produced by vegetable spores or animal parasites.

There is ground for suspicion that a part of the artificial butter is made from nauseating substances.

The possibility of injury to health from a carelessly-prepared artificial butter must not be neglected.

Dr. Thomas Taylor presented this aspect of the case to the Senate Committee.²

It has already been mentioned that in the earlier processes employed in the manufacture of artificial butter the stomachs of sheep and pigs were digested with the fats employed.

¹ Arbeiten a. d. Kaiserlichen Gesundheitsamte, pp. 494, 500.

² *Op. cit.* pp. 42-46 and 273-4.

Tidy and Wigner¹ have investigated the action of mammary tissue on fats used as butter substitutes.

By digesting a pure animal fat with the chopped-up tissues of the udders of cows the authors found a marked chemical change produced. Cleomargarine or tallow when treated in this way give rise to both soluble and volatile fatty acids. Since both milk and butter contain a certain amount of mammary tissue, in the form of casts from the mammary glands, it is believed that they also would exert an influence on animal fats. Butter appears to act more vigorously than milk in this way, probably because it contains a larger percentage of mammary tissue.

NUTRITIVE VALUES OF BUTTER AND OLEOMARGARINES.

On this subject Atwater² has collected valuable information, he says:

The value of butter, as of any other food material for nourishment, depends upon the amounts of its nutritive ingredients, their digestibility, and their uses in the nutrition of the body.

CHEMICAL COMPOSITION.

The food values of real and imitation butter, as compared with each other and with other food materials, can be best shown by first comparing their composition.

It appears that the nutrients of the leaner kinds of meat and fish consist mostly of protein, that the fatter meats and fish contain considerable fat with the protein, that the vegetable foods have for the most part very little fat, and abound especially in carbohydrates, while the nutriments of butter and oleomargarine consist almost exclusively of fats. Indeed, the protein and carbohydrates in both must be regarded as impurities. The quantities of fat are shown by analysis to be very nearly the same in both.

DIGESTIBILITY.

Regarding the relative digestibility of butter and oleomargarine the experimental facts at hand are meager. They imply, as would be expected from the composition, that there is very little difference between the two. The study of the question is rendered difficult by the fact that what is ordinarily called the digestibility of a food includes several different things, the ease with which it is digested, the time required for digesting it, and the proportions of its several constituents that are digested.

As to the comparative ease and time of digestion of butter and oleomargarine nothing is definitely known, though there is little ground for assuming that, in the alimentary canal of a healthy person, at any rate, one would be digested and taken into the circulation much more readily than the other. The actual amounts digested are capable of more nearly accurate experimental estimate. During the past few years very many experiments have been made, in Germany especially, to test the quantities of the more important constituents of different foods digested by domestic animals, and a considerable number have been carried out with men and children.

The only comparative experiments on the digestibility of butter and oleomargarine that have been reported are two series conducted by Professor Mayer, a German chemist. One series was with a full-grown man and the other with a boy of nine years of age, both strong, healthy persons. The outcome was that both the man and the boy digested from 97.7 to 98.4 per cent. of the fat of the butter, and from 96.1 to 96.3 per cent. of the fat of the oleomargarine. The average difference was about 1.6 per cent. in favor of the butter. There are, however, certain unavoidable sources of

¹ Analyst, 1883, pp. 113 *et seq.*

² Bradstreet's, Saturday, June 19, 1886.

error in such experiments, and it is very probable that the proportions actually digested were somewhat larger than these figures imply. Very likely each of the two persons may have digested practically all of the fat of the butter, and all but 1 or 2 per cent. or even less of that of the oleomargarine. In these experiments the butter and oleomargarine were eaten with bread, cheese, white of eggs, potatoes, peas, and sugar.

The digestibility of butter has been tested in two or three other series of experiments. Thus Dr. Rubner, in Munich, found that a healthy man, on a diet of butter, bread, and meat, digested 97.3 per cent. of the total fat of the food, of which the bulk came from the butter. In some experiments by myself, in which a man received a diet of fish (haddock) and butter, 91 per cent. of the total fat, nearly all of which came from the butter, was found to be digested.

The experiments of Rubner and myself were conducted in the same manner as those of Mayer, and exposed to the same slight sources of error. The results of all of them are just what would naturally be expected, namely, that very nearly all of the fat of butter and of oleomargarine is digested in a healthy organism.

It might seem that the relative digestibility of the two materials could be tested by experiments in artificial digestion; that is to say, by treating both substances with digestive fluid, or with materials similar to them, and observing the results. Such experiments are not accurate tests of the actual digestibility of the substances in the body, since the conditions which obtain in the alimentary canal cannot be exactly imitated by any artificial means which physiological chemistry has yet suggested. Professor Mayer, taking into account that the fats are more or less split up in the process of natural digestion, has made some experiments to test the comparative readiness with which butter and oleomargarine are split up, and finds a very slight difference in favor of butter. As the result of all his experiments he concludes that, while the butter appears to be a little more digestible than oleomargarine, the difference is too small to be of practical consequence for healthy persons. At the same time there may be cases, especially those of invalids and children just past the nursing period, when butter would be preferable; but, considering simply the nutritive values for ordinary use, Professor Mayer considers the choice between the two to be essentially one of comparative cost, an opinion from which there is, so far as I am aware, scarcely any dissent among those who have devoted the most study to this class of subjects.

It is a common and perhaps correct theory, though it lacks experimental confirmation, that the flavor of the fats peculiar to butter may in some way increase its value for nutriment. But, granting this to be true, it would be hardly reasonable to assume that a difference in flavor which even experts may fail to detect could make any considerable difference in the nutritive effect of two substances otherwise so similar as real and imitation butter.

To recapitulate briefly, butter and oleomargarine have very nearly the same chemical composition; in digestibility there may be a slight balance in favor of butter, though for the nourishment of healthy persons this difference can hardly be of any considerable consequence; for supplying the body with heat and muscular energy, which is their chief use in nutrition, they are of practically equal value, excelling in this respect all other common food materials. Such, at any rate, is the practically unanimous testimony of the latest and best experimental research.

While it is true that chemical analysis and certain digestive experiments have not hitherto shown that pure butter possesses any marked superiority over butter surrogates as a food, yet it must not be forgotten that butter has a much more complex composition than lard or tallow or cotton-seed oil; that it is a natural food, and doubtless possesses many digestive advantages which science has not yet been able to demonstrate.

THE MANUFACTURE OF ARTIFICIAL BUTTER IN THE UNITED STATES.

The following information has been kindly furnished by the Hon. Joseph S. Miller, Commissioner of Internal Revenue:

TREASURY DEPARTMENT, OFFICE OF INTERNAL REVENUE,
Washington, March 4, 1887.

SIR: In reply to your letter of 1st instant, I have the honor to state that there are thirty-seven factories engaged in the manufacture of artificial butter now in operation in the United States, located as follows:

Location.	No. of factories.	Location.	No. of factories.
Denver, Col.....	2	Buffalo, N. Y.....	1
Chicago, Ill.....	11	Columbus, Ohio.....	1
Kokomo, Ind.....	1	Cleveland, Ohio.....	3
Kansas City, Kans.....	1	Philadelphia, Pa.....	3
Armourdale, Kans.....	1	Pittsburgh, Pa.....	1
Cambridge, Mass.....	1	Allegheny, Pa.....	1
Brooklyn, N. Y.....	2	Providence, R. I.....	3
New York, N. Y.....	3	Pawtucket, R. I.....	2

There are two hundred and fifty-nine wholesale dealers in the United States, located as follows:

Location.	No. of wholesale dealers.	Location.	No. of wholesale dealers.
Birmingham, Ala.....	2	Salt Lake, Utah.....	1
Fort Smith, Ark.....	1	Hoboken, N. J.....	1
Hot Springs, Ark.....	1	Jersey City, N. J.....	1
Little Rock, Ark.....	2	Deming, N. Mex.....	1
Pine Bluff, Ark.....	1	Fairbanks, Ariz.....	1
Denver, Col.....	4	Saratoga Springs, N. Y.....	1
Jacksonville, Fla.....	1	Rochester, N. Y.....	1
Atlanta, Ga.....	1	Youngstown, Ohio.....	2
Englewood, Ill.....	1	Philadelphia, Pa.....	12
Springfield, Ill.....	1	Allegheny City, Pa.....	1
Cairo, Ill.....	2	Woonsocket, R. I.....	1
Council Bluffs, Iowa.....	1	Memphis, Tenn.....	8
Elwood, Kans.....	1	El Paso, Tex.....	2
Louisville, Ky.....	3	Dallas, Tex.....	1
New Orleans, La.....	5	Dennison, Tex.....	1
Boston, Mass.....	30	Milwaukee, Wis.....	3
Fall River, Mass.....	1	Ashland, Wis.....	1
Lowell, Mass.....	3	Eau Claire, Wis.....	1
Aspen, Col.....	1	Worcester, Mass.....	3
Pueblo, Col.....	1	Houghton, Mich.....	1
Durango, Col.....	1	Detroit, Mich.....	6
Buena Vista, Col.....	1	Grayling, Mich.....	1
Hartford, Conn.....	2	Saginaw, Mich.....	1
New Haven, Conn.....	4	Luddington, Mich.....	1
Leadville, Col.....	4	Saint Paul, Minn.....	2
Chicago, Ill.....	21	Helena, Mont.....	2
Peoria, Ill.....	1	Jefferson City, Mont.....	1
Danville, Ill.....	1	South Butte, Mont.....	1
Indianapolis, Ind.....	1	Ogden, Utah.....	1
Kansas City, Kans.....	2	Dover, N. H.....	1
Topeka, Kans.....	1	Newark, N. J.....	1
Covington, Ky.....	1	Santo Fé, N. Mex.....	1
Baltimore, Md.....	2	Albuquerque, N. Mex.....	1
Salem, Mass.....	1	New York, N. Y.....	15
New Bedford, Mass.....	2	Buffalo, N. Y.....	1
Gloucester, Mass.....	2	Cincinnati, Ohio.....	6
Lawrence, Mass.....	2	Cleveland, Ohio.....	3
Springfield, Mass.....	1	Pittsburgh, Pa.....	14
Ironwood, Mich.....	1	Providence, R. I.....	16
Bay City, Mich.....	3	Pawtucket, R. I.....	1
East Saginaw, Mich.....	1	Nashville, Tenn.....	1
Muskegon, Mich.....	1	San Antonio, Tex.....	1
Grand Rapids, Mich.....	4	Fort Worth, Tex.....	2
East Saint Louis, Ill.....	1	Richmond, Va.....	1
Butte, Mont.....	2	Oshkosh, Wis.....	1
Missoula, Mont.....	1	Hurley, Wis.....	2
Omaha, Nebr.....	3	Chippewa Falls, Wis.....	1

The quantity manufactured and removed for consumption or sale at 2 cents per pound during the months of November and December, 1886, and January, 1887, is as follows:

	Pounds.
November	4,742,569
December.....	2,786,278
January	2,501,114
Total.....	10,029,961

The quantity exported from the United States during the period above, all exportations being from the port of New York, is as follows:

	Pounds.
November	3,247
December.....	58,689
January	52,761
Total.....	114,697

Respectfully,

JOS. S. MILLER,
Commissioner.

Hon. N. J. COLMAN,
Commissioner of Agriculture, Washington, D. C.

COLORING MATTERS IN BUTTER.

The pure animal fats, prepared in the manner described, are almost colorless. The tint of genuine butter is imparted to these bodies by various coloring matters. The principal artificial colors which have been employed are:

- Annotto (*Bixa orellana*).
- Turmeric (*Curcuma longa* and *viridiflora*).
- Saffron (dried stigmas *Crocus sativus*).
- Marigold leaves (*Calendula officinalis*).
- Yellow wood (*Morus tinctoria*).
- Carrot juice (*Daucus carota*).
- Chrome yellow (PbCrO_4).
- Dinitrocressol—kalium.

ANNOTTO.

This substance is used more than any other in imparting to artificial butter a yellow tint. Indeed it is used to color genuine butter, which often in winter is almost white in its natural state.

The coloring substance called *annotto*, *arnatta*, or *roucou* is the reddish pulp surrounding the seeds in the fruit of *Bixa orellana*, a middling-sized tree growing in Guiana and other parts of South America. The pulp is separated by bruising the fruit, mixing it with water, then straining through a sieve, and allowing the liquid to stand till the undissolved portion subsides. The water is then poured off and the mass which remains, having been sufficiently dried, is formed into flat cakes or cylindrical rolls and sent into the market. Another mode is to bruise the seeds, mix them with water, and allow the mixture to ferment. The coloring matter is deposited during the fermentation, after which it is removed and dried. In commerce there are two kinds of annotto the Spanish or Brazilian and French, the former coming in

baskets from Brazil, the latter in casks from French Guiana. The French, which is also called *flag annotto*, has a disagreeable smell, probably from having been prepared by the fermenting process, but is superior as a dye-stuff to the Spanish, which is without any disagreeable odor. Annatto is of a brownish red color, usually rather soft but hard and brittle when dry, of a dull fracture, of a sweetish peculiar odor, and a rough, saline, bitterish taste. It is inflammable, but does not melt with heat. It softens in water, to which it imparts a yellow color, but does not dissolve. Alcohol, ether, the oils, and alkaline solutions dissolve the greater part of it. It contains a peculiar crystallizable coloring principle, to which M. Preisser, its discoverer, gave the name of *bixin*. It is frequently adulterated with red ochre, powdered bricks, colcothar, farinaceous substances, chalk, sulphate of calcium, turmeric, &c. The mineral substances, if present, will be left behind when the annatto is burned.¹

SAFFRON.

Saffron has a peculiar, sweetish, aromatic odor, a warm, pungent, bitter taste, and a rich deep orange color, which it imparts to the saliva when chewed. The stigmas of which it consists are an inch or more in length, expanded and notched at the upper extremity, and narrowing towards the lower, where they terminate in a slender, capillary, yellowish portion, forming a part of the style. When chewed it tinges the saliva deep orange-yellow. Saffron should not be mixed with the yellow styles. When pressed between filtering paper it should not leave an oily stain. When soaked in water it colors the liquid orange-yellow, and should not deposit any pulverulent mineral matter nor show the presence of organic substances differing in shape from that described.²

Adulteration of saffron.—Saffron is often adulterated with cheaper yellow vegetable coloring matter, turmeric, annatto, the flowers of the marigold (*Calendula officinalis*), Carthamus flowers, the flowers of *Arnica montana*, *Scolymus hispanicus*, *Pulicaria dysenterica*, *Punica granatum*, *Pæonia*, *Crocus vernus*, &c.³

Of these the marigold flowers are perhaps the most commonly used. They have a natural yellow color, and when they are saturated with carmine or aniline red, and dried, they possess a striking similarity to the genuine saffron. If they are put for a few minutes in water, however, they assume their original form, and are then easily distinguished from the stigmas of the saffron flower.

If a mixture of saffron stigmas and the substitutes just mentioned be put into a vessel of water where the individual pieces are widely separated, the saffron stigmas soon become surrounded with a yellow extract, while the others suffer no change or impart only a weak carmine tint to the water.

The use of mineral coloring matters like the chromate of lead is highly reprehensible from a sanitary point of view.

Annatto and saffron in butter may be detected by the following method, proposed by Cornwall:⁴

About 5 grams of the warm filtered fat are dissolved in about 50cc. of ordinary ether, in a wide tube, and the solution is vigorously shaken for ten to fifteen seconds with 12 to 15cc. of a very dilute solution of caustic potash or soda in water, only alkaline enough to give a distinct reaction with turmeric paper, and to remain alkaline after separating from the ethereal fat solution. The corked tube is set aside and in a few

¹ U. S. Dispensatory, p. 1572.

² U. S. Dispensatory, p. 501.

³ Schimpfer, Anleit. z. Mikroskopischen Untersuchung d. Nahrungs- und Genussmittel, p. 101.

⁴ Chem. News, vol. 55, p. 49.

hours, at most, the greater part of the aqueous solution, now colored more or less yellow by the annatto, can be drawn from beneath the ether with a pipette or by a stopcock below, in a sufficiently clear state to be evaporated to dryness and tested in the usual way with a drop of concentrated sulphuric acid.

Sometimes it is well to further purify the aqueous solution by shaking it with some fresh ether before evaporating it, and any fat globules that may float on its surface during evaporation should be removed by touching them with a slip of filter-paper; but the solution should not be filtered, because the filter-paper may retain much of the coloring matter.

The dry-yellow or slightly orange residue turns blue or violet blue with sulphuric acid, then quickly green, and finally brownish or somewhat violet (this final change being variable, according to the purity of the extract).

Saffron can be extracted in the same way; it differs from annatto very decidedly, the most important difference being in the absence of the green coloration.

Genuine butter, free from foreign coloring matter, imparts at most a very pale yellow color to the alkaline solution; but it is important to note that a mere green coloration of the dry residue on addition of sulphuric acid is not a certain indication of annatto (as some books state) because the writer has thus obtained from genuine butter, free from foreign coloring matter, a dirty green coloration, but not preceded by any blue or violet blue tint.

Blank tests should be made with the ether; it is easy to obtain ether that leaves nothing to be desired as to purity.

Turmeric is easily identified by the brownish to reddish stratum that forms between the ethereal fat solution and the alkaline solution before they are intimately mixed. It may be even better recognized by carefully bringing a feebly alkaline solution of ammonia in alcohol beneath the ethereal fat solution with a pipette, and gently agitating the two, so as to mix them partially.

Martin¹ gives a method of separating and determining artificial coloring matters in butter. To 5 grams of fat, dry, are added 25cc. CS₂ and the mixture well shaken with water made slightly alkaline with NaOH or KOH and the mixture gently shaken. The alkaline water will dissolve all the coloring matter. This is now determined qualitatively by the spectroscope or quantitatively by making up a comparative mixture with the coloring matter found. Butters act better when treated as above than oleomargarine.

The relative amount of color in butters is thus estimated by Babcock:²

The relative amount of color in butters may be determined with accuracy as follows: One gram of the fresh butter is digested with 15cc. of refined kerosene till the fats are completely dissolved and the solutions filtered. The filtrate will be colored in proportion to the coloring matter of the butter, and may be compared to that from another butter or preferably to a standard solution by means of a Dubesque colorimeter. A standard color for comparison may be prepared by adding a small quantity of any of the commercial butter colors to kerosene oil. This standard will keep for a long time without changing, if kept from the light.

The scale of the colorimeter on the side which the butter solution occupies is always set at the same degree, while the scale for the other standard is made variable. The reading of this side will, therefore, vary with the amount of color in the sample.

If some of the kerosene oil in which the butters are dissolved be substituted for the solution of butter, a small reading will be obtained which should be deducted

¹ Analyst, 1885, p. 163.

² Fifth An. Rep't B'd Control; N. Y. Exp. Sta., p. 335-336.

from that for each of the butters. The numbers remaining are directly proportional to the colors of the butters. In the butters thus far examined a fair colored Jersey butter was taken for a standard and called 100. The others were calculated to this standard from the scale reading.

The use of a small amount of vegetable coloring matters mentioned above does not seem to be prejudicial to health.

EXAMINATION OF BUTTERS.

The examination of butters to detect adulterations may be divided into two parts: (1) Determination of physical properties; (2) determination of chemical properties.

Physical properties.—The physical properties of fats which are useful in butter analysis are their crystalline state, specific gravity, and melting point.

Pure fresh butter prepared in the ordinary manner is not crystalline. The microscope shows the absence of all forms of crystalline structure, and thin films of the butter fat have no influence whatever on polarized light.

On the contrary, old butters, or butters which have been melted and allowed to crystallize, and oils and fats which have been once in a fluid state, show, as solids, quite a distinct crystalline structure readily revealed by the microscope and affecting, in a marked manner, the polarized ray.

Recently much attention has been excited by a discussion of the application of polarized light to the qualitative examination of suspected butters, and since many analysts have not the time to fully investigate this matter I have thought it useful to enter upon the discussion of it in considerable detail.

Polarization is a term applied to a phenomenon of light, in which the vibrations of the ether are supposed to be restricted to a particular form of an ellipse whose axes remain fixed in direction. If the ellipse becomes a straight line it is called "plane polarization." This well-known phenomenon is most easily produced by a Nicol prism, consisting of a crystal of carbonate of calcium (Iceland spar). This rhombohedral crystal, the natural ends of which form angles of 71° and 109° , respectively, with the opposite edges of its principal section, is prepared as follows:

The ends of the crystal are ground until the angles just mentioned become 68° and 112° . The crystal is then divided diagonally at right angles with the planes of the ends and with the principal section, and after the new surfaces are polished they are joined again by Canada balsam. The principal section of this prism passes through the shorter diagonal of the two rhombic ends. If now a ray of light fall on one of the ends of this prism, parallel with the edge of its longer side, it suffers double refraction, and each ray is plane polarized, the one at right angles with the other. That part of the entering ray of light

which is most refracted is called the ordinary and the other the extraordinary ray. The refractive index of the film of balsam being intermediate between those of the rays, permits the total reflection of the ordinary ray, which, passing to the blackened sides of the prism, is absorbed. The extraordinary ray passes the film of balsam without deviation and emerges from the prism in a direction parallel with the incident ray, having, however, only half of its luminous intensity.

Two such prisms, properly mounted, furnish the essential parts of a polarizing apparatus. They are called the "polarizer" and the "analyzer," respectively.

If now the plane of vibration in each prism be regarded as coincident with its principal section, the following phenomena are observed: If the prisms are so placed that the principal sections lie in the prolongation of the same plane, then the extraordinary polarized ray from the polarizer passes into the analyzer, which practically may be regarded in this position as a continuation of the same prism. It happens, therefore, that the extraordinary polarized ray passes through the analyzer exactly as it did through the polarizer, and is not reflected by the film of balsam, but emerges from the analyzer in seemingly the same condition as from the polarizer. If now the analyzer be rotated 180° , bringing the principal section again in the same plane, the same phenomenon is observed. But if the rotation be in either direction only 90° , then the polarized ray from the first prism, incident on the second, departs itself exactly as the ordinary ray, and on meeting the film of balsam is totally reflected. The field of vision, therefore, is perfectly dark.

In all other inclinations of the planes of the principal sections of the two prisms the ray incident in the analyzer is separated into two, an ordinary and extraordinary, varying in luminous intensity in proportion to the square of the cosine of the angle of the two planes.

Thus by gradually turning the analyzer, the field of vision passes slowly from maximum luminosity to complete obscurity.

The expression "crossed Nicols" refers to the latter condition of the field of vision.

Selenite plate.—In the practical application of polarized light to the examination of facts, an important use is made of a selenite plate (crystallized sulphate of calcium). A disk of selenite, interposed between the polarizer and analyzer imparts a coloration to the field of vision which varies with the relative position of the principal sections of the two prisms.

This phenomenon depends on the fact that a plane polarized ray of light can be decomposed, in passing a section of a bi-refracting crystal like selenite or mica, into two rays, polarized at right angles and differing in phase.

This fact is illustrated by passing a polarized ray (from a Nicol prism) through a very thin crystallized plate of mica or gypsum (selenite) obtained by cleavage. By the double refraction of the thin plate the po-

larized ray is separated into two, ordinary and extraordinary. The extraordinary, having to pass over a greater distance, joins the ordinary ray, after emergence, with a phase slightly different, the degree of difference depending on the nature of the lamina, the inclination of the incident ray, &c., but in every case this difference of phase can be easily calculated, and the resultant beam of light is said to be elliptically polarized. Each of the components of this ray enters the analyzer and is again resolved. One of its elements is suppressed in the Nicol and the other, consisting of vibrations in the principal plane, passes through. The result is two sets of vibrations in the same plane slightly different in phase, which are, therefore, in a condition to interfere and produce color. If the source of light be monochromatic, when the analyzer is rotated, only certain variations in luminous intensity will be observed; but if, on the other hand, white light be employed these variations in phase will give rise to a display of colors. In order that the field of vision be of a uniform tint it is necessary that the lamina of crystal be of uniform thickness. For ordinary use the selenite plate is ground to a thickness which will give green and red tints.

For crossed Nicols the colors of the selenite plate appear brightest when it is so placed that the plane of vibration in the crystal forms an angle of 45° with the plane of vibrations of the polarized incident ray. If the selenite plate is rotated in its own plane, the color appears in the four quadrants at its maximum and disappears at intervals of 90° .

If the planes of the two Nicols are parallel, the same order of phenomena appear as before, except that the positions of maximum and minimum are reversed.

If the analyzer be rotated and the selenite plate and polarizer remain stationary there is no effect produced, when the principal section of the selenite is parallel or perpendicular to the polarizing plane of the under Nicol. But if this plane is inclined less than 45° to that of the polarizer, then the selenite plate in a complete revolution of the analyzer will appear four times brightly colored and four times colorless. In adjoining quadrants the colors will be complementary. When the Nicols are so placed as to produce the maximum intensity of color, if small bi-refracting crystals be introduced at random into the field of vision, they will, in general, have the same effect on the plane polarized ray as the selenite plate. Since the axes of these crystals may have any accidental position with reference to the planes of the Nicols, it follows that the field of vision, which before appeared of a uniform tint, will now become variegated, the color disappearing in some cases and becoming more intense in others.

When a bi-refracting crystal is cut into laminae normal to its axis, of appropriate thickness, it gives some peculiar phenomena when examined with polarized light. When the analyzer is perpendicular to the polarizer, there is seen in the ordinary image a black cross, the existence of which can be explained by the mathematical theory of polar-

ization. The arms of this cross are parallel and perpendicular to the primitive plane of polarization. Between the arms are generally to be found rings which present the successive tints of the fringes of interference. In the extraordinary image the order of the phenomenon is entirely reversed.

Having now briefly described the more important optical phenomenon which forms the basis of the examination of butters with polarized light, I will next say something of the nature of the substances to be examined.

The expressions "fats" and "oils" designate those natural products of animals and vegetables known as glycerides. Chemically considered they are the normal propenyl ethers of the fatty acids, or, in other words, compounds of the triad alcohol, glycerine, with the fatty acids. The term "fat" is applied to such bodies when they are solid at ordinary temperatures, and "oil" when they are semi-solid or liquid. Those which are most important are:

Tri-stearin, $C_3H_5(C_{18}H_{35}O_2)_3$, occurs in natural fats. It may be obtained in a considerable degree of purity by repeated crystallizations from ether. It crystallizes in plates of a pearly luster. Its melting point is $55^\circ C$.

Tri-palmitin, $C_3H_5(C_{16}H_{31}O_2)_3$, is found in animal fats and palm oil. It crystallizes with a pearly luster from ether. The crystals have a melting point of from 50° to $66^\circ C$.

Tri-butylin, $C_3H_5(C_4H_7O_2)_3$, occurs chiefly in butter. At ordinary temperature it is liquid, and has a distinct and peculiar odor and taste.

Tri-olein, $C_3H_5(C_{18}H_{33}O_2)_3$, occurs in animal fats and in almond and olive oil. At ordinary temperatures it is liquid, is neutral to test papers, and has neither taste nor smell.

Minute quantities of tri-myristin, caprin, caprylin, and caproin are also found in butter.

Pure butter fat is supposed to contain—

	Per cent.
Tri-olein, about	42.5
Tri-stearin, about	51.0
Tri-butylin, about	6.3
Other glycerides, about2
	<hr/> 100.00

Olive oil is composed chiefly of tri-palmitin and olein.

Tri-stearin is the chief constituent of mutton fat, it having only small quantities of olein and palmitin.

Beef fat has somewhat more palmitin and stearin than mutton tallow. Lard has more olein.

It is thus seen that in dealing with butter fats and their substitutes we have to consider chiefly tri-olein and stearin, and, in smaller quantities, tri-palmitin, butylin, &c. It follows, therefore, that the chief differences in the several substances will be due to the different proportions

in which these glycerides are mixed and to such other physical differences as the various sources of the substances under examination would produce. These differences, however, prove greater when subjected to physical and chemical analysis than the foregoing résumé of their chemical constitution would indicate. Advantage has been taken of these differences of physical structure to discriminate between fats and oils of different origins. The specific gravity and the melting point furnish two valuable points of discrimination, but both of these are perhaps inferior in value to the evidence afforded by the crystalline structure of the fats. The observation with the microscope of the crystals obtained in various ways furnishes valuable data for discrimination, and if the light employed be plane polarized or elliptically polarized by a selenite plate, these data become still more valuable.

The first account of the use of the selenite plate in such examinations was given by Dr. J. Campbell Brown in the *Chemical News*, vol. 28, pages 1, *et seq.* He gives the following directions for the polaro-microscopic work :

Examine several portions of the original sample by means of a good microscope, using a one-fourth or one-fifth inch object-glass. In butter made from milk or cream nothing is seen except the characteristic globules, and the granular masses of curd and the cubical crystals of salt. The hard fats of butter are present in the globules in a state of solution, and are not recognizable in a separate form.

If stearic acid, stearin, or palmitin be present in separate form, they will be recognizable by simple fusiform crystals, or starlike aggregations of acicular crystals. They indicate the presence of melted fats.

Other substances, such as starch flour, palm oil, corpuseles, Irish moss, coloring matter, &c., may also be distinguished by the microscope as distinct from butter or fats.

Examine the same portions with the same object-glass, together with a polariscope, consisting of two Nicol's prisms and a selenite plate. The crystals referred to polarize light, and when viewed by the polariscope are distinctly defined. Particles of suet and other fats which have not been melted may also be distinguished by their action on polarized light, by their amorphous form, and by their membranes.

The value of this department of fresh-butter fat with elliptically polarized light did not meet with the appreciation its merits deserved until attention was again called to it by Prof. Thomas Taylor, of the Department of Agriculture.

Any fat or oil which is homogeneous and non crystalline will present the same phenomena when viewed with polarized light and selenite plate; in other words will have no effect on the appearance of the field of vision. It is only, therefore, fats which are in a crystalline or semi-crystalline state that can thus be distinguished from fresh, amorphous butter. Naturally it follows that a butter which has been melted and cooled, or butter which has stood a long time, would impart a mottled appearance to the field of vision. For a simple preliminary test, however, the procedure is worthy of more attention than its discoverer, Dr. J. Campbell Brown, accorded to it.

FORMS OF FAT CRYSTALS.

The forms of fat crystals differ greatly with the kinds of fat and the proportions in which they are mixed. It would be idle to attempt a description of all these modifications.

Husson¹ has published an illustrated description of some of the more important fat crystals. Suet crystals, according to Husson, are very characteristic of stearin. They are small, rounded, or elliptical masses formed by stiff, needle-like crystals, and resemble a sea urchin or hedge hog.

In lard are seen polyhedral cells arising from the compression of the fatty globules. In impure lard are also seen the remains of cells and adipose tissue. Fresh butter shows some long and delicate needles of margarine (?) united in bundles and grouped in various ways. When the butter is melted these needles diminish in length and become grouped round a central point. I have mentioned these descriptions especially for the purpose of calling attention to the fact, that, in the illustrations of the microscopic appearance of butter and other fats, emphasis is often given to one particular phenomenon, and the real appearance as seen in the microscope is not reproduced.

The only reliable representation is found in the actual photo-micrograph or its exact graphic reproduction.

When the crystals of certain fats are prepared in a special way they show, with polarized light, a distinct cross, the existence of which is explained by the laws of elliptical polarization already mentioned.

This cross was first described by Messrs. Hefner and Angell in 1874 in the following words:

If some of a fat containing crystals be placed on a slide and a drop of castor or olive oil be applied and pressed out with a thin glass cover, the depolarization of light is much enhanced; a revolving black cross, not unlike that on some starch grains, is seen in great perfection. These crosses are most clearly defined in the crystals obtained from butter, and these thus mounted form a brilliant polariscopic object.

They add further:

Thus far and no farther, as it seems to us, can the microscope assist us in this matter; but even such indications are valuable, especially when subsequent analysis proves the sample to be an adulterated article. The microscopic evidence in such case frequently serves to clinch together the whole superstructure, and thus certainty is made doubly sure.

Dr. Thomas Taylor has further called attention to this phenomenon in a paper read before the American Society of Microscopists at its Cleveland meeting, August, 1885. On page 3 of the reprint of this paper he says:

Since the publication of that paper I have experimented largely with butter, and have made the discovery that when it is boiled and cooled slowly for a period of from twelve to twenty-four hours at a temperature of from 50° to 70° Fahr. it not only becomes crystallized, but, with proper mounting and the use of polarized light, it ex-

¹ Ann. d. Chem. et d. Pharm., vol. 5, pp. 12, 469.

hibits on each crystal a well-defined figure resembling what is known as the cross of Saint Andrew. In course of time, the period ranging from a few days to a few weeks according to the quality of the butter used and the temperature to which it is exposed, the crystals, which at first are globular, degenerate, giving way to numerous rosette-like forms peculiar to butter.

On page 5 he says :

About ten years ago, while making some experiments with boiled butter, I first observed it exhibited small crystals somewhat stellar in form, but gave no further attention to the fact until May last. For the purpose of determining the real form of the crystal of boiled butter I procured a sample of pure dairy butter from Ohio. I boiled it, and when cold examined it under a power of 75 diameters. To my surprise I found globular bodies. When I subjected them to polarized light a cross consisting of arms of equal length was observed on each crystal. On rotating the polarizer the cross of each crystal rotated. On rotating the glass on which the specimen of butter was mounted the crosses remained stationary, thus showing that the appearance of the cross depends, probably, on the fact that the crystals are (1) globular, (2) polarizing bodies, (3) translucent, and (4) comparatively smooth. Were they opaque or non-polarized or did they consist of long spines, causing great divergence of the rays of light, no image of the cross would be visible; showing that the appearance of the cross under polarized light and the conditions stated is not due to any physical structure of the fatty crystals themselves. But from whatever cause the appearance of the cross on the butter crystals arises, its constant appearance on new butter under the conditions above described is a fact beyond any question; and, as far as my experience goes, the better the quality of the butter the more clearly defined is the cross; it is black, large, and well defined. When these crystals are under polarized light and a selenite plate combined they exhibit the prismatic colors, but the cross proper is not visible in this case, although the crystals are still divided into four equal parts and are exceedingly interesting objects.

Dr. Taylor having thus directed the attention of scientists to these important phenomena, it has not taken long to show that there is little reason for the rather mean opinion of European chemists of the value of the microscope in detecting adulterations of butter. In several cases of prosecution before the District authorities the offenders have been convicted solely on the microscopical evidence and have admitted the justice of the sentence. If only fresh butters were exposed for sale, and all adulterants were certainly once melted and slowly cooled, but little more than this qualitative examination would be necessary.

Prof. H. A. Weber, of Columbus, Ohio, has made some interesting experiments with the microscope on fats, which in the main bear out the conclusions of Messrs. Brown, Helmer and Angell, and Taylor. As was to be expected, however, he has shown that the appearance of the cross on a crystal of natural fat does not show that it is derived from pure butter. He says, in Bulletin No. 13 of the Ohio Experimental Station, Experiments 7, 8, 9, and 10 :

Experiment 7.—The difference between the behavior of the tallow fats in Experiment 3 and the last three experiments could only be ascribed to a difference of conditions. It is well known that table butter normally contains 4 to 6 per cent. of salt and 5 to 20 per cent. of water. These ingredients constitute the most marked difference between butter and the rendered animal fats as tallow and lard. In order to test the effect of this mixture upon the tallow fats, about half an ounce of the oleo oil used in Experiment 3 was mixed in a porcelain mortar with a small quantity of

salt and eight or ten drops of water. After the water was thoroughly incorporated, the mass was transferred to a test tube and boiled for 1 minute as in the case of butter. It was then poured into a wooden pill-box and allowed to cool as before. The cooled mass presented quite a marked difference in appearance from that obtained from the same substance in Experiment 3. It retained to a great extent the yellow color of the oleo oil, was of a more granular nature, and in fact resembled boiled butter in every respect. When a small particle was stirred up with olive oil on a glass slide it separated readily. When covered and viewed with a pocket lens it revealed a mass of globules resembling insect eggs. Under the microscope these globules exhibited essentially the same characteristics as those obtained from butter in Experiment 1. The crystalline mass of the oleo globule seemed somewhat coarser, and to this condition was ascribed the fact that the cross, as well as the colors produced by the selenite plate, were less sharply defined than in the globules obtained from butter. The slides prepared from this material were remarkably free from the small detached crystals of fat observed in Experiment 3.

Experiment 8.—Having thus discovered that these globular masses may be obtained from pure tallow fat by simply observing the conditions which obtain in butter making, the following test was made: Nine grams of oleo oil and 1 gram of lard were placed in a small beaker glass and eight or ten drops of a saturated solution of salt in water added. The mixture was then gently heated to melt the fats. After shaking violently for a few moments to mix the salt solution with the fats, the mixture was boiled gently for 1 minute and then allowed to cool as before in a wooden pill-box. The microscopic examination of this preparation revealed globular masses which could in no wise be distinguished from those obtained from pure butter. The crystalline texture was dense, the cross of St. Andrew's plainly marked, and the colors produced by the selenite sharply defined.

Experiment 9.—A mixture of one part of lard to five parts of oleo oil was treated as in the last experiment with like results.

Experiment 10.—In this test a mixture consisting of 20 per cent. of lard and 80 per cent. of oleo oil was employed. Whether the consistency of this mixture was peculiarly adapted to the formation of the globules, or whether possible variations of conditions in manipulation were more favorable, the writer is unable to judge from a single experiment, but the fact is that in this case the individual "butter crystals" were exceedingly large and characteristic.

The use of polarized light in photo-micrography is also valuable in enabling the photographer to print the light-colored crystals on a dark background. To illustrate some of the forms of crystals of butter and its substitutes as they appear under polarized light a large number of microscopic samples were prepared and photographed by Messrs. Richards and Richardson. Results of some of the more interesting of these photographs are herewith transmitted. In all cases the figures are magnified 40 diameters, unless otherwise stated.

DESCRIPTION OF PLATES.

PLATE I.

FIG. 1.—Fresh butter boiled.

FIG. 2.—Fresh butter made in the laboratory without the use of salt, melted, filtered, and boiled. A small sample of the butter was taken and boiled for one minute in a test tube over the naked flame, then set aside and allowed to cool slowly for twenty-four hours. A suitable quantity was then taken, sufficient to make a slide for the microscope, thinned with olive oil, and pressed out on the cover.

PLATE II.

FIG. 3.—Fresh Virginia butter boiled and let stand for seven days. Specimen prepared by Dr. T. Taylor, March 11, 1886.

FIG. 4.—Fresh Virginia butter boiled. Specimen prepared by Dr. T. Taylor March 11, 1886.

PLATE III.

FIG. 5.—Fresh Kentucky butter boiled. Specimen prepared by Dr. T. Taylor March 11, 1886.

FIG. 6.—Fresh butter boiled. Specimen prepared by Dr. T. Taylor March 15, 1886.

PLATE IV.

FIG. 7.—Filtered butter fat dissolved in boiling alcohol and allowed to cool slowly.

FIG. 8.—Filtered butter fat dissolved in boiling ether and allowed to cool slowly. The fresh butter was melted and filtered through a jacketed filter, thus getting rid of the water, curd, and salt; allowed to cool and prepared as above.

PLATE V.

FIG. 9.—Beef suet fat. Not boiled. The suet fat was cut up into fine pieces and melted in the water bath at a low heat and filtered; allowed to cool slowly. Specimen was taken several days after the sample was prepared.

FIG. 10.—Beef suet fat boiled with the addition of salt and cooled slowly.

PLATE VI.

FIG. 11.—Beef suet fat, "oleo oil," dissolved in boiling ether and allowed to cool slowly.

FIG. 12.—Beef suet fat, "oleo oil," dissolved in boiling alcohol and allowed to cool slowly.

PLATE VII.

FIG. 13.—Leaf lard. Not boiled. Specimen taken direct from can as purchased in the open market. Magnified 160 diameters.

FIG. 14.—Lard dissolved in boiling ether and allowed to cool slowly.

PLATE VIII.

FIG. 15.—Beef fat, "oleo oil," and lard, "neutral," boiled with salt and water and allowed to cool slowly.

FIG. 16.—Butterine, from Armour & Co., Chicago. Boiled and allowed to cool slowly.

PLATE IX.

FIG. 17.—Butterine, from Armour & Co., Chicago. Not boiled. Specimen taken direct from tub as received July, 1886. Magnified 160 diameters.

FIG. 18.—Butterine, from Armour & Co., Chicago. Melted, filtered, and boiled.

PLATE X.

FIG. 19.—Butterine, from Armour & Co., Chicago. Dissolved in boiling ether and allowed to cool slowly.

FIG. 20.—Oleo oil, from Armour & Co., Chicago. Melted, filtered, and boiled. Allowed to stand four days under cover glass.

PLATE XI.

FIG. 21.—Oleomargarine, from Armour & Co., Chicago. Not boiled. Specimen taken direct from tub as received July, 1886. Magnified 160 diameters.

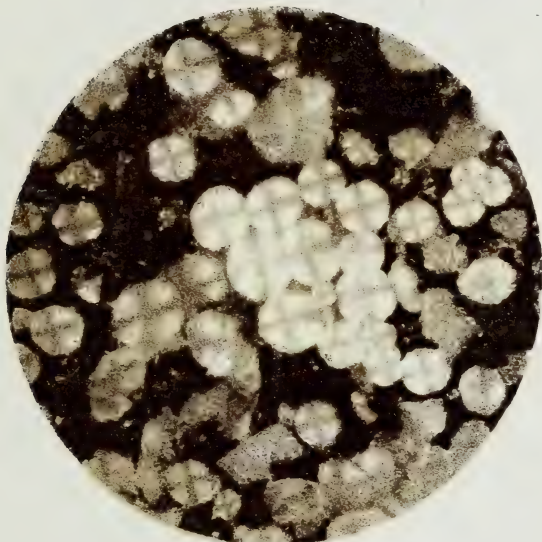
FIG. 22.—Oleomargarine, from Armour & Co., Chicago. Boiled with salt and water and allowed to cool slowly.

PLATE XII.

FIG. 23.—Same as Fig. 22.

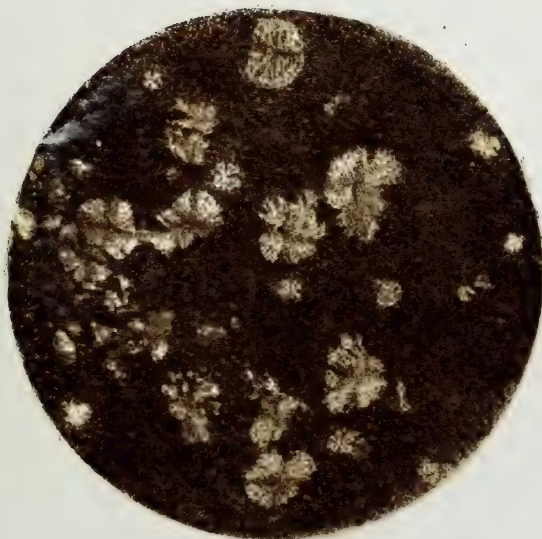
FIG. 24.—Same as Fig. 22.

Fig 1



BUTTER x40

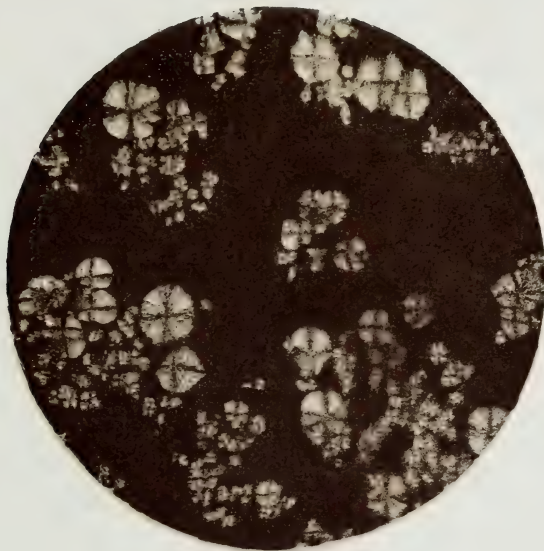
Fig 2



BUTTER x40

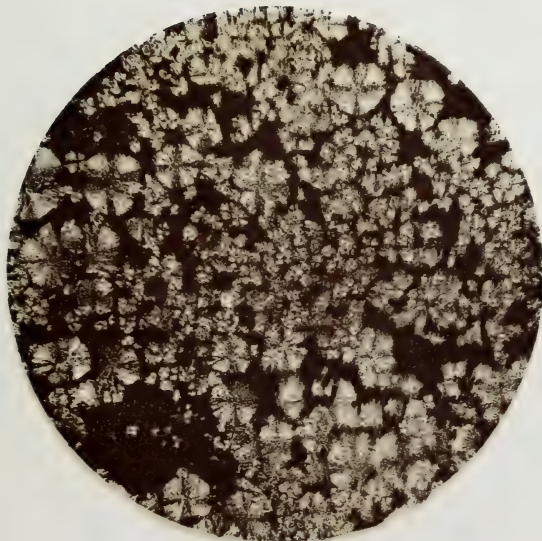


Fig 3



BUTTER x40

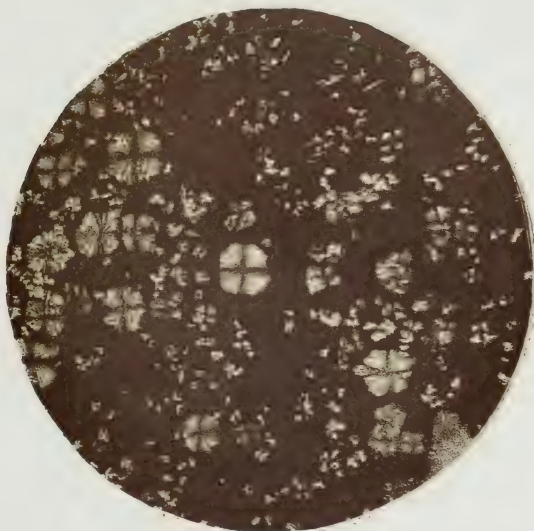
Fig 4



BUTTER x40

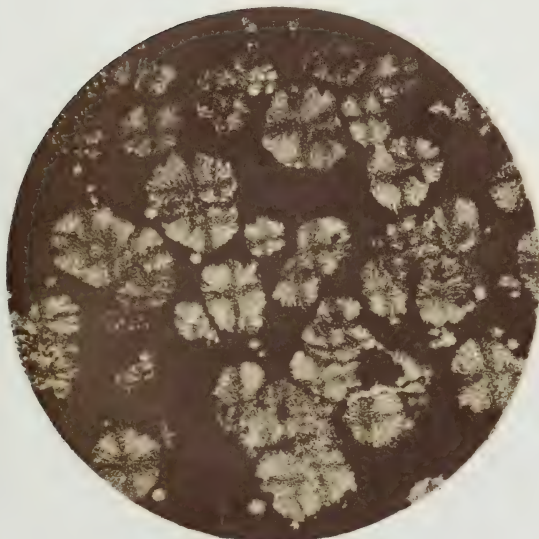


Fig 5



BUTTER x40

Fig 6



BUTTER x40

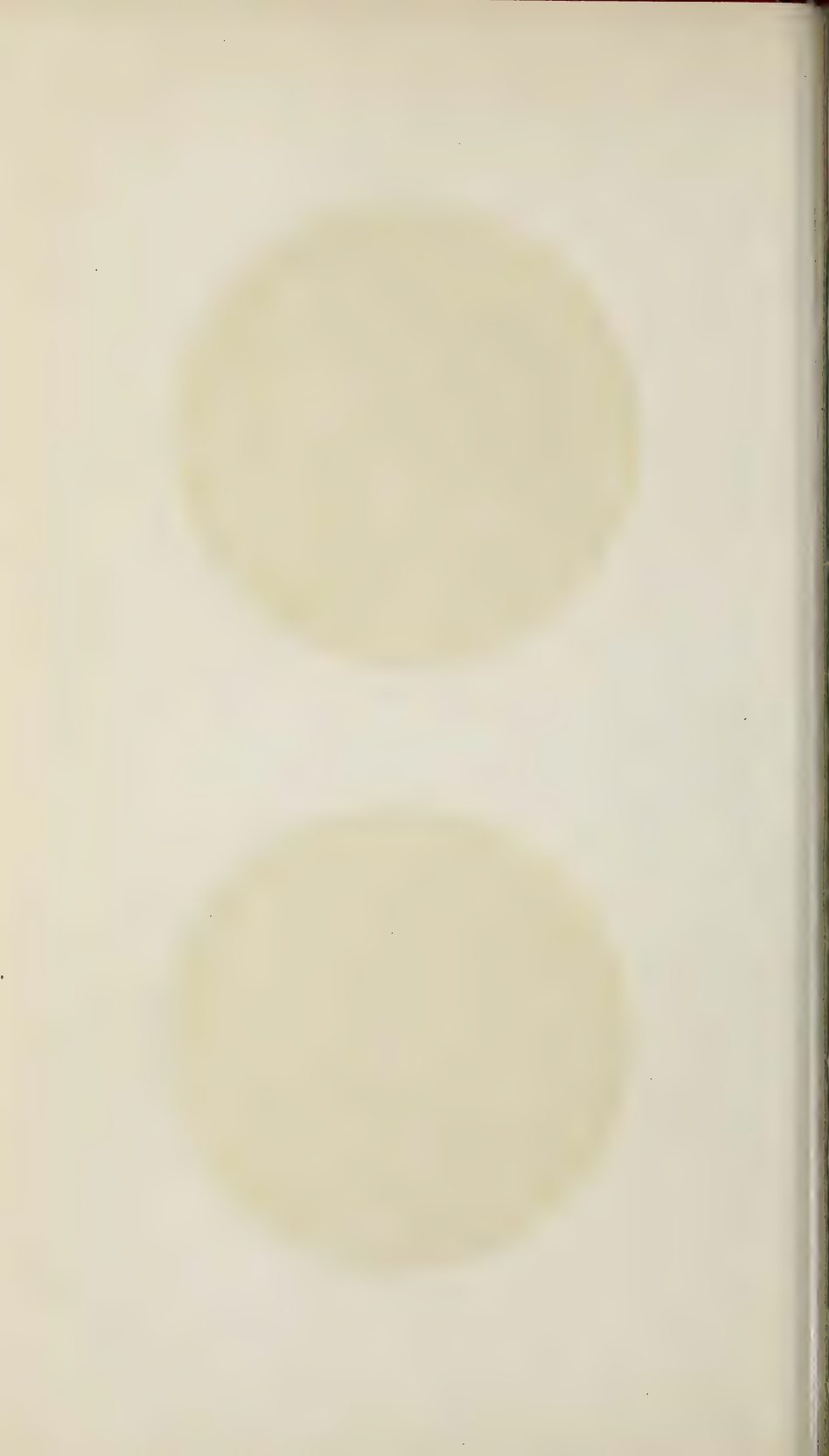
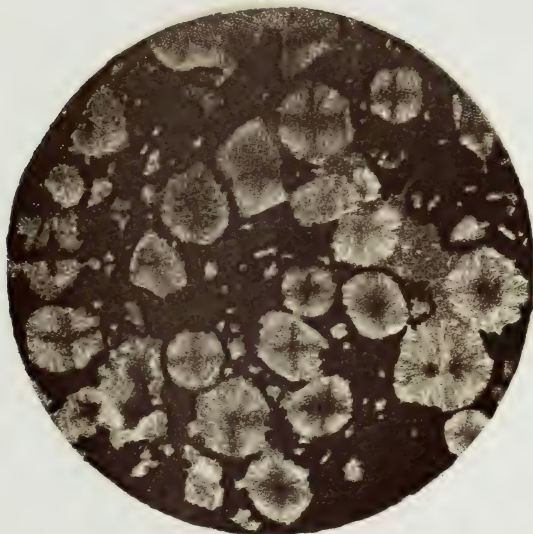
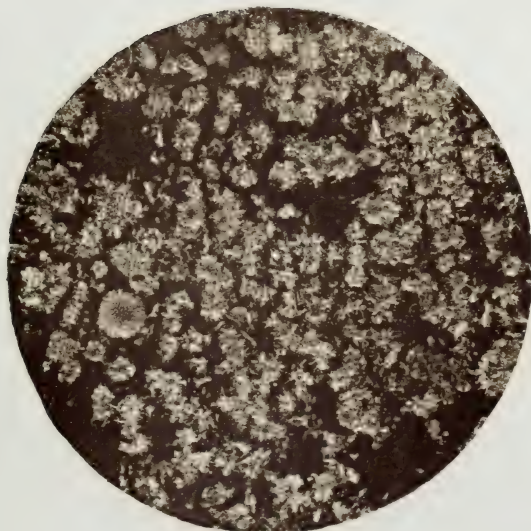


Fig 7



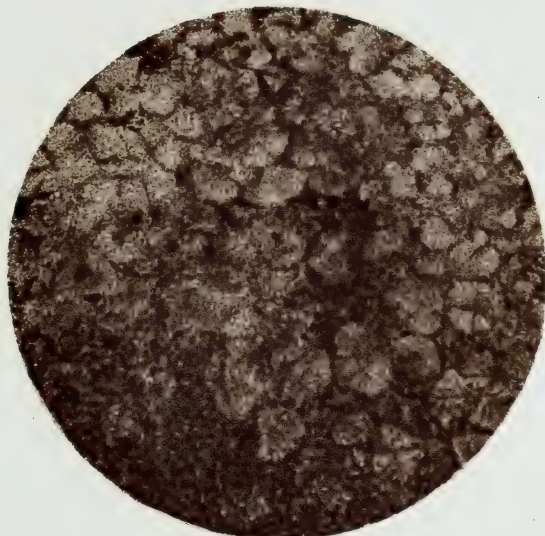
BUTTER x40

Fig 8



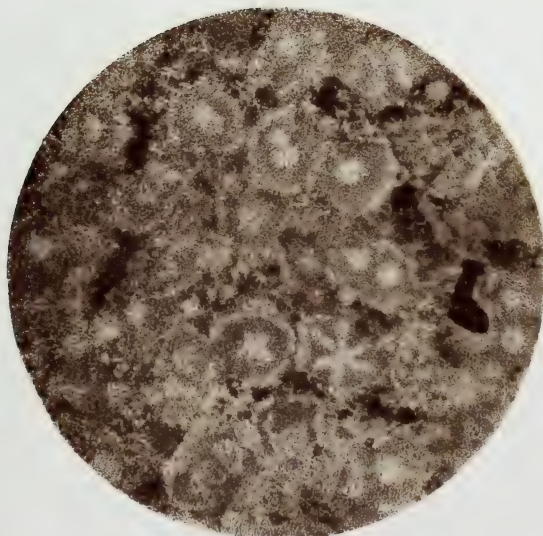
BUTTER x40

Fig 9



BEEF FAT x40

Fig 10



BEEF FAT x40

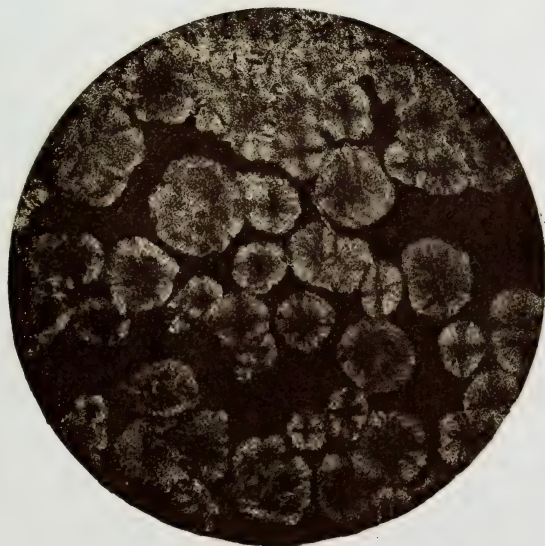


Fig 11



BEEF FAT x40

Fig 12



BEEF FAT x40

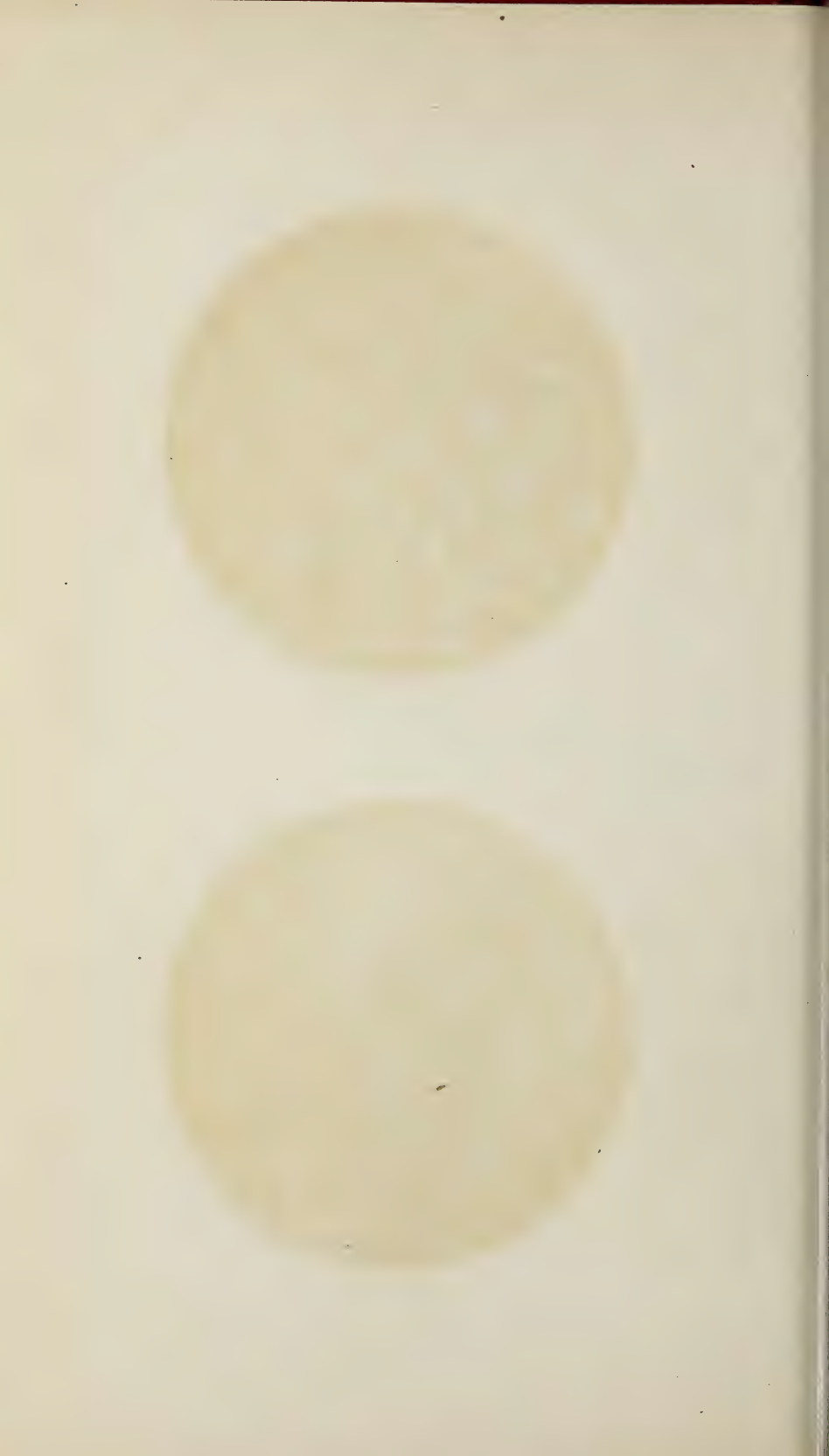
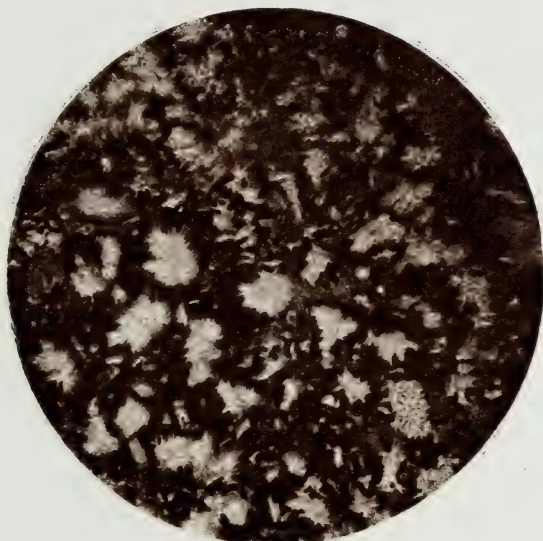
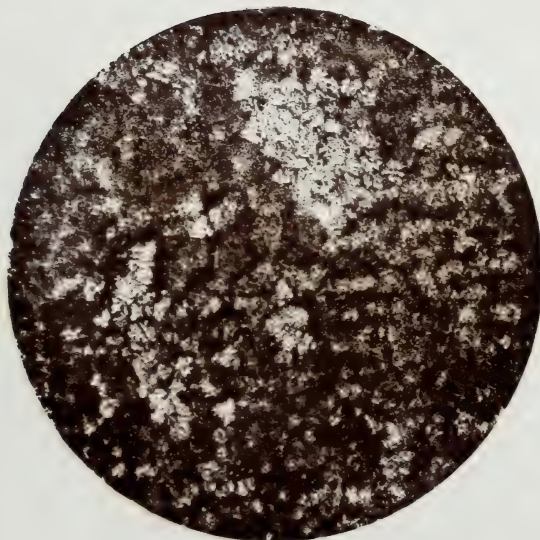


Fig 13



LARD x160

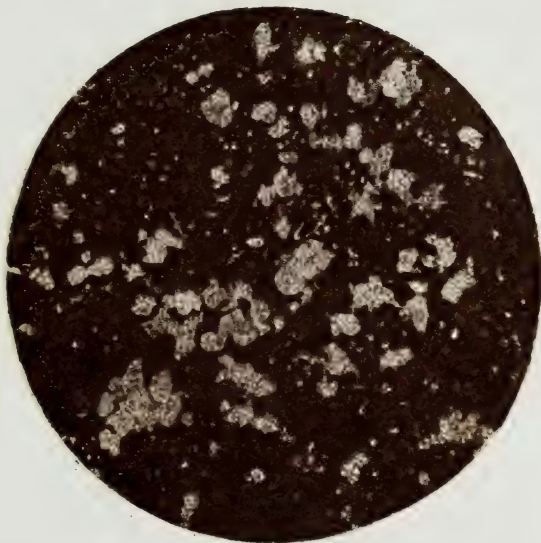
Fig 14



LARD x40

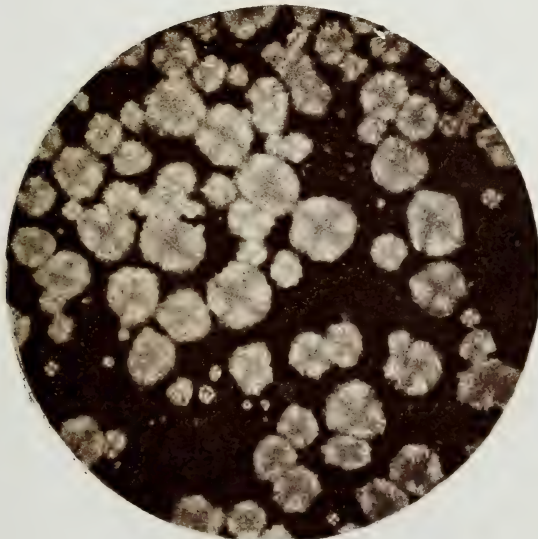


Fig 15



BEEF FAT & LARD x40

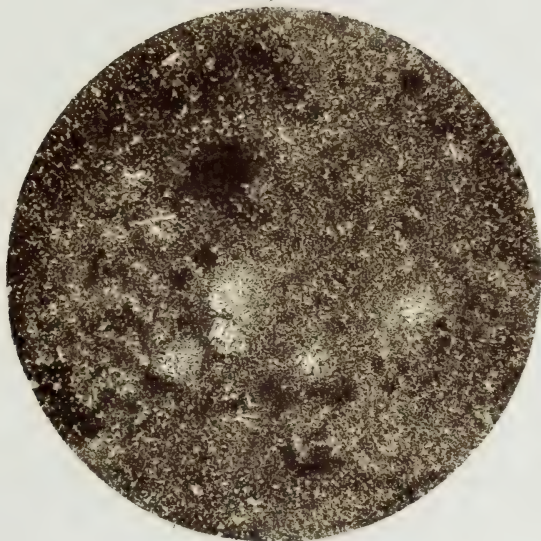
Fig 16



BUTTERINE x40



Fig 17



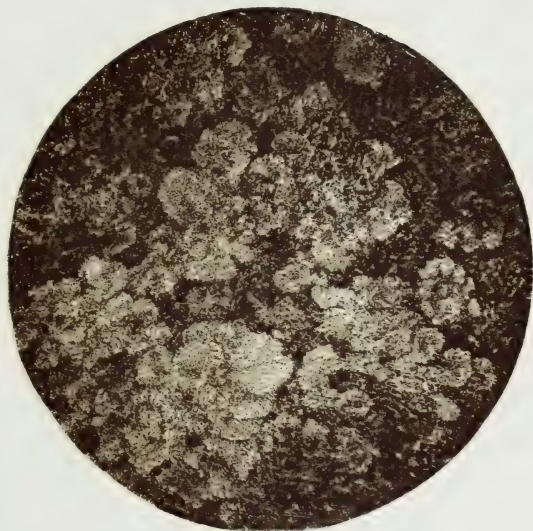
BUTTERINE x160

Fig 18



BUTTERINE x40

Fig 19



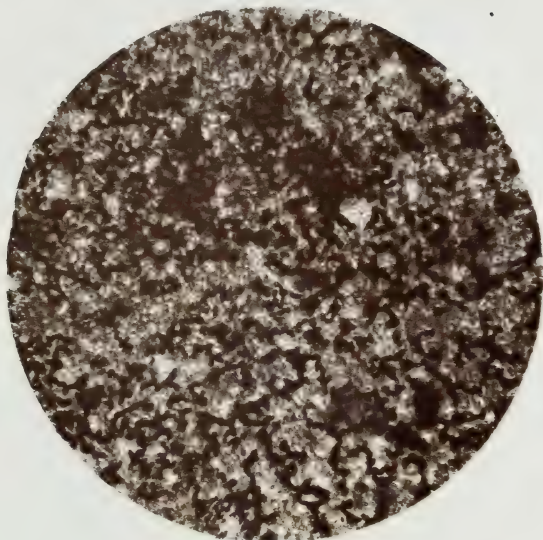
BUTTERINE x40

Fig 20



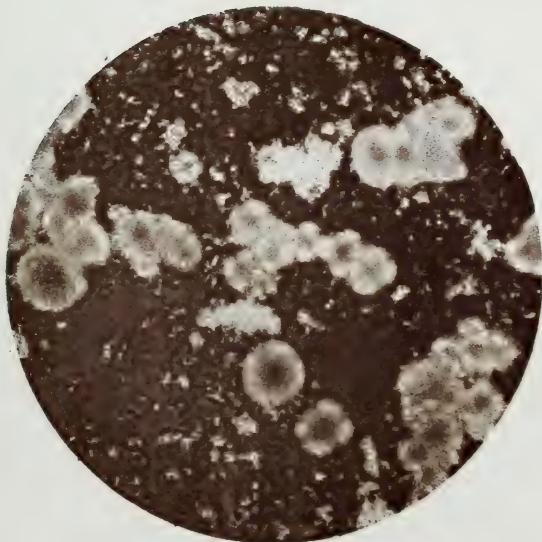
BEEF FAT x40

Fig 21



OLEOMARGARINE x160

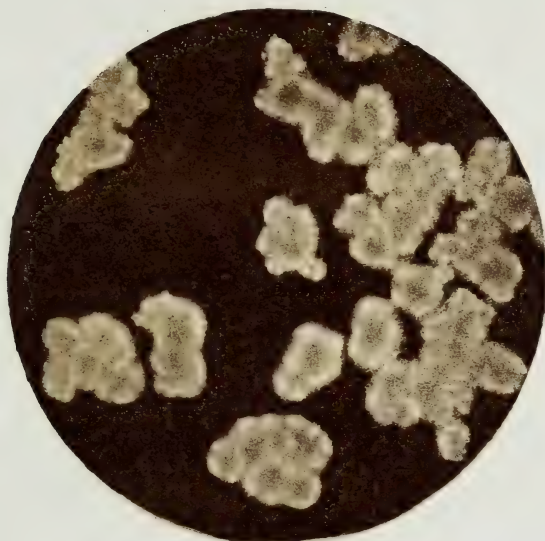
Fig 22



OLEOMARGARINE x40

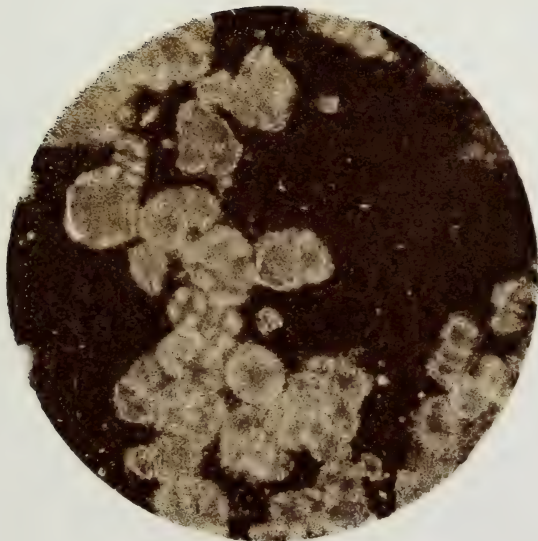


Fig 23



OLEOMARGARINE x40

Fig 24



OLEOMARGARINE x40



A careful study of these illustrations will show that the microscope and polarized light are most valuable and reasonably certain means whereby a qualitative examination of butters can be made. The approximate amount of added fat can only be determined by a chemical analysis of the suspected sample, taken in connection with some of its further physical characteristics.

I have given above all the really valuable points heretofore established in respect of the use of polarized light in butter and fat analyses.¹

The use of the microscope in butter examinations has not commanded as much attention among analysts as its merits deserve. Sell says:²

Though investigations have shown that the differences in structure under the microscope are not in all cases sufficiently characteristic to determine a sharp distinction between two different fats, yet it must be admitted that the microscopic examination is able to prove the presence of foreign fats at the moment it succeeds in establishing the presence of molecular tissues in animal or vegetable parasites.

Leudtner and Hilger say:³

The use of the microscope in the examination of fats requires a still further development before it can become generally applicable.

Having written to the editor of the Analyst for some information on the subject of the use of the microscopic methods in England, he replied: "The whole subject has been studied over and there is nothing in it."

Dr. S. M. Babcock, chemist of the New York Experimental Station, says:⁴

At the time these butters were received there was considerable controversy regarding the efficiency of Dr. Taylor's method for the detection of adulterations in butter by means of the microscope. An excellent opportunity was offered in these samples for testing this method in an impartial manner, and a microscopical examination of them was made before the nature of the butters was revealed by other tests. The butters were examined directly with polarized light and a selenite plate, and afterwards the crystals from the melted butters were examined in the same way for the "Saint Andrew's cross."

The direct examination with polarized light and a selenite plate showed prismatic colors in all of the adulterated butters, and a uniform tint in all of the genuine butters, except No. 2, which appeared very much like the adulterated samples. The crystals from all of the butters, adulterated as well as genuine, gave a well-defined Saint Andrew's cross with polarized light. This was also the case with neutral lard (No. 14), in which the cross was sharply defined, though quite small. No. 15 consisted of stearine from the oil-oil factories, and showed no cross when examined by itself, but when combined with a small quantity of butter fat the crystals formed had the same appearance as those from pure butter.

The method has also been quite unsatisfactory in trials made at the station with butters whose character was known. Whether these results were due to a lack of skill or to imperfect knowledge in the details of the work I do not know. The

¹ Notices of minor importance on the same subject can be found in Chem. News, vol. 4, pp. 230, 283, 309, 322; Zeit. Anal. Chem., 1872, p. 334; Journ. Royal Microscopical Society, 1873, p. 378; American Quarterly Microscopical Journal, 1878, p. 294; American Journal of Microscopy, October, 1878; Bied. Centralblatt, 1879, pp. 861-865; 1882, p. 345; 1882, p. 49; Amer. Chemist, vol. 2, p. 428.

² *Op. cit.*, p. 503.

³ Ver. Bay. Vertreter d. Angewand. Chem., p. 222.

⁴ Fifth Ann. Rept. Bd. Control N. Y. Exp. Sta., pp. 330, 331.

uncertain results of some skilled microscopists, however, would indicate that the difficulty is inherent in the method. It certainly is not simple, and is not calculated to supersede the chemical methods now in use.

Caldwell,¹ after references to the notices of the use of the microscope in the examination of butter published up to that time (1882), says, p. 519:

It is plain, therefore, that little dependence can be placed on any microscopic test of the genuineness of butter, at least so far as the observation of the crystalline forms of foreign fats is concerned, for neither does the absence of such forms prove that the butter does not contain oleomargarine, nor does their presence prove the adulteration.

On the other hand, Mylius² has shown that the polarization microscope may be used for the detection of minute quantities of foreign fats in butter. Pure butter gives with crossed Nicols a dark field, whereas crystals of foreign fat will appear bright. Skalweit³ recommends this method highly, and affirms that even the kind of foreign fat present may be determined.

In spite of the generally unfavorable opinions I feel sure that the chemist who neglects to make a simple microscopic examination of a suspected butter with polarized light and a selenite plate loses a valuable qualitative indication of the character of the samples with which he has to work. The melting of the sample of butter and its slow cooling to secure good bi-refracting crystals I consider a much less valuable indication than the simple observations above described.

SPECIFIC GRAVITY.

The determination of the specific gravity of a butter fat gives a most valuable indication of its purity. The density of pure butter glycerides is distinctly greater than that of the common adulterants, with the exception of cotton-seed oil. While this difference is not great, it is nevertheless large enough to be easily detected by careful manipulation.

Manipulation.—The relative weight of the filtered and dried fat is to be determined in a pycnometer. This flask should be carefully calibrated by weighing the pure distilled water it will contain at the temperature at which the subsequent determinations are to be made. The flask should be provided with a delicate thermometer, but this is not essential, since the temperature can be determined by an external thermometer.

The temperature at which the determinations should be made is evidently that at which all the common butter adulterants will be in a perfectly fluid state. Generally the temperature of 100° F. has been employed. Since, however, "neutral lard" may have a melting point as high as 40° C. or even a little above that I have uniformly taken the specific gravity at that degree. In case the fat should have a melting point a little above this the temperature can be raised until the fat is

¹ Second Ann. Rept. N. Y. S. Bd. of Health.

² Correspondenzblatt des Vereins Anal. Chem., 1878, No. 3.

³ *Ibid.*, 1879, Nos. 5 and 13.

fluid and can then be reduced to 40°C ., without danger of solidification. The difference between the specific gravities expressed at 37°C . and 40°C . is not of very great magnitude.

Blyth¹ recommends the use of a pycnometer of 50 to 100 grams capacity, with a thermometer stopper. This is filled with water at 35°C . and placed in a beaker of water at 43°C . When the water has reached a temperature of $37^{\circ}.7\text{C}$. the flask is removed and weighed.

The fat whose density is to be determined is treated in the same manner and weighed at the same temperature.

Wigner² places the butter-fat in a wide tube where a bubble of the specific gravity of .896 is kept below the surface by the bulb of the thermometer. At a certain temperature the bubbles will slowly sink to the bottom. In butters of .911 density, above which a sample may be passed as pure, these beads will sink as follows:

Specific gravity of beads.....	.889	.8896
Temperature.....	$62^{\circ}.7\text{C}$.	$55^{\circ}.5\text{C}$.

If the beads sink at any temperature lower than these the butter will need further examination.

Estcourt³ describes a method of taking specific gravity of fats as follows:

The bulb of a Westphal balance is suspended in a test-tube containing the fat, the test-tube being immersed in paraffin, in a water bath. The adjustment of the weights takes place at a temperature of $92^{\circ}.2\text{C}$. This process has been modified by König.⁴

In König's process there are several water baths which are closed with the exception of a tube for carrying off the steam. In the cover of each bath are four openings for the reception of four test-tubes $1\frac{1}{4}$ inches in diameter and 8 to 9 inches long. These are fastened air-tight into the openings mentioned. Each tube stands one-half inch above the cover of the water bath. Each piece of apparatus when in use contains in one of the tubes a sample of pure butter and in the others the samples under examination.

The specific gravity is determined by small areometers 6 inches long and with a scale marked from .845 to .870. The numbers obtained at 100°C . were as follows: Pure butter, .867; artificial butter, .859; beef fat, .860; mutton fat, .860; lard, .861; horse fat, .861. Mixture of pure butter with other fats gave numbers between .859 and .865.

The process of König has been tested by the Board of Health at Berlin and found relatively useful.⁵ The method has also been approved by Elsner;⁶ by Ambuhl and Dietzsch;⁷ and Meyer.⁸

¹ Foods, p. 295.

² Blyth, *op. cit.*, p. 295.

³ Chem. News, vol. 34, p. 254.

⁴ Industrieblätter, 1879, p. 455.

⁵ Sell, *op. cit.*, p. 505.

⁶ Die Praxis des Nahrungsmittelchemikers, 2d ed., p. 50.

⁷ Rep. d. Ver. Anal. Chem., 1884, p. 359.

⁸ Zeit. Anal. Chem., 1881, p. 376.

Jones¹ calls attention to the fact that the specific gravity of butter increases with age.

The specific gravities of several samples are compared in the following table, the numbers in second column being obtained after eighteen months :

Specific gravity at 37.7 C.

Sample.	1.	2.
1.....	.9123	.9083
2.....	.9105	.9114
3.....	.9119	.9185
4.....	.9112	.9165
5.....	.9125	.9155
6.....	.9133	.9132

In other samples there was a decrease in specific gravity. In five samples out of nine there was an increase and the percentage of soluble acids had also increased.

Since butters in general are obtained for analysis without having been long kept the observation of Jones does not have much practical importance.

Sendtner and Hilger² find that a filtered pure butter fat does not show a specific gravity less than .866 at 100° C. In the Erlangen University numerous experiments with twenty different samples of butter showed variations from .866 to .8685.

Allen³ recommends Sprengel's tube for the determination of specific gravity of oils at the temperature of boiling water.

The weight of the Sprengel tube and that of water contained in it at 15° 5 C. being known, the tube should be completely filled with the oil by immersing one of the orifices in the liquid and gently sucking the air from the other orifice of the tube. The tube is then placed in the mouth of a conical flask containing water kept in a rapid ebullition, and the cover of a porcelain crucible placed over it. As the oil gets hot it expands and is expelled in drops from the horizontal capillary orifice of the tubes. When the expansion ceases any oil adhering to the orifice is removed by cautious application of filter paper, the tube removed from the bath, wiped dry, allowed to cool, and weighed. The weight of the contents divided by the weight of water at 15° 5 C. previously known to be contained by the tube will give the density of the oil at the temperature of the boiling water; water at 15° 5 C. being taken as unity.

Bendikt⁴ prefers the Sprengel tube to all the other methods of estimating the specific gravity of oils. He also recommends the Westphal balance as used by Bell and Walkenhaar.

¹ Analyst, 1879, p. 39.

² Vereinbarungen betreffs d. Untersuch. u. Beurteilung v. Nahrungs-Genussmitteln, pp. 221-2.

³ Com. Organic Analysis, Vol. 2, 2d ed., p. 15.

⁴ Analyse der Fette, &c., p. 53.

Dr. Muter¹ gives the following table of the specific gravity of various oils at 37° C.

Kind of oil.	Specific gravity.	Kind of oil.	Specific gravity.
Olive oil	907.0	Linseed oil (boiled)	938.0
Almond oil	905.6	Castor oil	955.8
Arachis oil	908.5	Sperm oil	872.4
Rape oil	906.7	Whale oil	906.0
Nut oil	908.4	Seal oil	915.0
Cotton-seed oil (brown)	917.6	Codliver oil	917.9
Cotton-seed oil (refined)	913.6	Lard oil	907.8
Poppy-seed oil	915.4	Neat's-foot oil	907.0
Hempseed oil	919.3	Butterine	903 to 906.0
Linseed oil (raw)	925.2	Butter-fat	912 to 914.0

METHOD EMPLOYED IN CHEMICAL DIVISION.

When convenient the determination of specific gravity is not made until a number of samples is on hand. Each determination is made in duplicate. The pycnometers, holding about 25 grams, are filled with the filtered fat, at as low a temperature as possible, and placed in a flat dish filled with water as nearly to the tops of the flasks as possible. The pycnometers used should all be of the same height. The stopper has a capillary perforation for the escape of the oil as the temperature rises. If the pycnometers are not furnished with thermometers of their own, a delicate thermometer is suspended in the water surrounding them. The water-bath is slowly warmed and gently but constantly stirred until the temperature reaches 40° C. It is kept at this temperature for fifteen or twenty minutes, until the fat has taken on the same temperature as the water. The pycnometers are then carefully cleaned and dried, and, after cooling to the temperature of the balance-room, are weighed. This method is somewhat tedious when only one determination is to be made, but where many samples are to be examined it is sufficiently speedy. In respect of accuracy it leaves nothing to be desired.

TEMPERATURE AT WHICH SPECIFIC GRAVITY IS STATED.

Different analysts select different temperatures for determining specific gravity. It would be well to have some agreement on this point to avoid confusion.

Since the specific gravity determined at any temperature can be easily calculated for any other given temperature, I suggest that it might be well to express all specific gravities in terms of water at 4° C.

THE MELTING POINT OF FATS.

The fats pass rather slowly from the semi-solid state, which is their natural condition at ordinary temperatures, to complete fluidity. It is, therefore, difficult to determine accurately the exact temperature at which they melt.

¹ Allen, *op. cit.*, p. 15, foot-note.

The value of the melting point in the examination of fats is at once apparent, provided it is possible to be assured that it represents a definite temperature which can be easily and accurately determined.

At a temperature of 40° C. pure butter fat has a specific gravity of .912, while the substitutes therefor, viz, lard, tallow, oleo-oil, neutral lard, &c., have specific gravities varying from .900 to .905. Yet even these small differences are extremely valuable in distinguishing the fats from each other.

The differences in melting points, when they can be accurately determined, will also prove helpful to the analyst. The usual methods employed to determine melting points have been based on the assumption that a fat becomes transparent at the moment it assumes the liquid state. Usually the fat is melted and placed in glass capillary tubes, and, after cooling, put into water near the bulb of a thermometer. The water is slowly warmed, and the moment the fat in the tube becomes transparent the reading of the thermometer is taken. A careful observer is able in this way to make multiple determinations which agree well together, but the readings of different persons are apt to vary greatly. Moreover, it is not the *melting* but the *transparent* point that is determined.

In 1883, at the Minneapolis meeting of the Association for the Advancement of Science, I described a method of determining the flowing point of a fat. The melted fat having been put into a small bent metallic tube, was, after cooling, placed in a bath of mercury. One arm of this U-tube was slightly longer than the other. The bent tube was immersed in the mercury until the longer arm was just below the surface. The fat in the tube was, therefore, subjected to a certain definite pressure from the mercury, due to the difference in length of the two arms. When the melted fat first appeared on the surface of the mercury, the thermometric reading was made. It is scarcely necessary to add that the bulb of the thermometer was wholly immersed in the mercury. Fairly good results were obtained by this method.

Another method, which gave rather good results, I tried at the same time. A thin film of fat was spread over the surface of the mercury and the temperature noted at which a platinum wire drawn through it left no trace. The solidifying point was determined in the same operation by observing where the wire left a mark. Various methods for determining the melting point of fats are given by Reichert.¹ The method preferred by him is a modification of Guichard's process,² in which the fat is forced out of the tube by a water pressure of a constant magnitude.

Dr. H. Kriess³ describes an apparatus for estimating the melting point by the completion of an electric circuit dependent on the melting of the fat used as an insulating material. A platinum wire, bent into the

¹ Zeit. Anal. Chem., 1885, pp. 11 *et seq.*

² *Ibid.*, 1883, p. 70.

³ Zeit. f. Instrumentenkunde, vol. 4, pp. 32, 33.

form of a small hook, is dipped into the melted fat, a portion of which adheres to it. This process is repeated until a sufficient insulation is produced. The fat-covered end of the wire is then dipped into a mercury cup, which contains also the bulb of the thermometer. The cup is placed in the electric circuit and the moment of contact is determined by the ringing of an electric bell. Thorough trial of this method convinced me that it was less accurate than any of those which have already been mentioned.

Realizing the importance of determining some definite point at which fats would assume a constant condition under the influence of temperature, I was led to select another physical aspect of fats, easily and certainly visible, which could be regarded as the melting point. This condition may be defined as the point at which the molecular attraction of the fat becomes greater than the molecular cohesion.

If a thin film of any fat be suspended in a liquid of equal specific gravity with it and this liquid be slowly warmed, a point will be reached at which the film will roll up and finally assume the form of a sphere. By imparting to the globule a gentle motion of rotation the observer is easily able to distinguish the moment when it becomes sensibly symmetrical. I use the following method and apparatus for applying this principle to the determination of the melting points of fats.¹ The apparatus consists of (1) an accurate thermometer for reading easily tenths of a degree; (2) a less accurate thermometer for measuring the temperature of water in the large beaker glass; (3) a tall beaker glass, 35cm. high and 10cm. in diameter; (4) a test tube 30cm. high and 3.5cm. in diameter; (5) a stand for supporting the apparatus; (6) some method of stirring the water in the beaker. I use a blowing bulb of rubber and a bent glass tube extending to near the bottom of the beaker; (7) a mixture of alcohol and water of the same specific gravity as the fat to be examined.

Manipulation.—The disks of the fat are prepared as follows: The melted and filtered fat is allowed to fall from a dropping tube from a height of 15 to 20cm. onto a smooth piece of ice floating in water. The disks thus formed are from 1 to 1½cm. in diameter and weigh about 200 milligrams. By pressing the ice under the water the disks are made to float on the surface, whence they are easily removed with a steel spatula.

The mixture of alcohol and water is prepared by boiling distilled water and 95 per cent. alcohol for ten minutes to remove the gases which they may hold in solution. While still hot the water is poured into the test-tube already described until it is nearly half full. The test tube is then filled with the hot alcohol. It should be poured in gently down the side of the inclined tube to avoid too much mixing. If the tube is not filled until the water has cooled the mixture will contain so many air bubbles as to be unfit for use. These bubbles will gather on

¹Journal Anal. Chemistry, vol. 1, No. 1, pp. 39 *et seq.*

the disk of fat as the temperature rises and finally force it to the top of the mixture.

The test tube containing the alcohol and water is placed in a vessel containing cold water, and the whole cooled to below 10° C. The disk of fat is dropped into the tube from the spatula, and at once sinks until it reaches a part of the tube where the density of the alcohol-water is exactly equivalent to its own. Here it remains at rest and free from the action of any force save that inherent in its own molecules.

The delicate thermometer is placed in the test tube and lowered until the bulb is just above the disk. In order to secure an even temperature in all parts of the alcohol mixture in the vicinity of the disk the thermometer is moved from time to time in a circularly, pendulous manner. A tube prepared in this way will be suitable for use for several days, in fact, until the air bubbles begin to attach themselves to the disk of fat. In no case did the two liquids become so thoroughly mixed as to lose the property of holding the disk at a fixed point, even when they were kept for several weeks.

In practice, owing to the absorption of air, I have found it necessary to prepare new solutions every third or fourth day.

The disk having been placed in position, the water in the beaker glass is slowly heated and kept constantly stirred by means of the blowing apparatus already described.

When the temperature of the alcohol-water mixture rises to about 6 degrees below the melting point, the disk of fat begins to shrivel, and gradually rolls up into an irregular mass.

The thermometer is now lowered until the fat particle is even with the center of the bulb. The bulb of the thermometer should be small, so as to indicate only the temperature of the mixture near the fat. A gentle rotary movement should be given to the thermometer bulb, and I have thought it would be convenient to do this with a kind of clock-work, although I have not carried this idea into execution. The rise of temperature should be so regulated that the last 2 degrees of increment require about ten minutes. The mass of fat gradually approaches the form of a sphere, and when it is sensibly so the reading of the thermometer is to be made. As soon as the temperature is taken, the test tube is removed from the bath and placed again in the cooler. A second tube, containing alcohol and water, is at once placed in the bath. It is not necessary to cool the water in the bath. The test tube (I use ice water as a cooler) is of low enough temperature to cool the bath sufficiently. After the first determination, which should be only a trial, the temperature of the bath should be so regulated as to reach a maximum about $1^{\circ}.5$ above the melting point of the fat under examination.

Working thus with two tubes about three determinations can be made in an hour.

After the test tube has been cooled the globule of fat is removed with a small spoon attached to a wire before another disk of fat is put in.

Agreement of multiple determinations.

FILTERED BUTTER FAT.

	Degrees C.
No. 1, by one observer.....	33.5
No. 2, by another	33.5
No. 3, by a third.....	33.9
No. 4, by a third.....	32.4
No. 5, by a third.....	34.4

A second set of observations made with the same butter gave—

	Degrees C.
No. 1	33.7
No. 2	33.8
No. 3	33.5
No. 4	33.5

A different butter gave the following numbers :

	Degrees C.
No. 1	34.0
No. 2	33.7
No. 3	33.8
No. 4	34.0

Another butter, "Creamery Tub," gave the numbers below :

	Degrees C.
No. 1	33.7
No. 2	33.7
No. 3	33.6
No. 4	33.6

A neutral lard, from Armour & Co., Chicago, gave the following results :

	Degrees C.
No. 1	42.8
No. 2	42.4
No. 3	42.3
No. 4	42.6
No. 5	42.2
No. 6	42.0

An oleo oil, from Armour & Co., gave—

	Degrees C.
No. 1	29.4
No. 2	29.5
No. 3	29.5
No. 4	29.7
No. 5	30.0
No. 6	30.3
No. 7	29.7
No. 8	29.8

Another butter, shown by the microscope to be adulterated, gave—

	Degrees C.
No. 1	33.5
No. 2	33.7
No. 3	33.5

No. 1777, a doubtful butter gave—

	Degrees C.
No. 1	34.3
No. 2	34.5
No. 3	33.6
No. 4	34.0

No. 1779, also a doubtful butter, gave—

	Degrees C.
No. 1	34.2
No. 2	33.5
No. 3	33.0

These results show that the method is capable of general application.

Collecting together the mean results obtained with butter-fats the following table is obtained :

TABLE No. 1.—*Melting points, etc., of genuine butter.*

Serial number.	Melting point.	Per cent. soluble acid.	Specific gravity at 40° C.
	° C.		
1745	34.5	5.48	.911
1766	34.3	4.52	.910
1768	34.2	5.21	.910
1769	33.7	5.05	.912
1772	34.0	5.26	.911
1785	32.0	4.48	.912
1786	34.7	4.32	.912
Mean	33.8	4.86	.911

TABLE No. 2.—*Melting points, &c., of butters of doubtful purity.*

Serial number.	Melting point.	Per cent. Soluble acid.	Specific gravity at 40° C.
	° C.		
1777	34.1	3.92	.910
1779	33.6	3.16	.909
1780	34.4	3.02	.910
1781	34.5	3.97	.910
Mean	34.1	3.51	.909

The above were all bought as pure butters. They are condemned on account of the low percentage of soluble acid, while by their specific gravity they appear to fall near the limit of purity. The soluble acid in the above was determined by washing out and not by Reichert's method.

TABLE No. 3.—*Melting point of substances sold as butter, but proved by analysis to be adulterated.*

Serial number.	Melting point.	Per cent. soluble acid.	Specific gravity at 40° C.
	° C.		
1755	39.0	1.53	.906
1778	33.6	0.21	.904
1787	34.6	0.09	.906
4594	35.3	0.09	.904
4595	37.8	0.90	.905

TABLE No. 4.—*Melting point, &c., of "oleo-oil" and "neutral lard" used as butter adulterants.*

Serial number.	Melting point.	Per cent. soluble acid.	Specific gravity at 40° C.
	° C.		
"Neutral lard":			
1754	42.4	0.10	.904
4597	42.4		
"Oleo-oil: "			
1756	29.7	0.08	.903
4596	29.6	0.08	.903

TABLE No. 5.—*Melting point of mixtures made in laboratory as indicated.*

[The butter used had a melting point of 33° 1 C.; the "oleo-oil" of 29° 6 C.; and the "neutral lard" of 42° 4 C.]

No.	Composition of mixture.	Theoretical melting point.	Observed melting point.
		° C.	° C.
1....	2 parts butter, 1 part "neutral"	36.2	35.2
2....	1 part butter, 2 parts "neutral"	39.3	39.6
3....	1 part butter, 1 part "neutral" and 1 part "oleo"	35.0	35.5
4....	2 parts butter, 1 part "oleo"	31.9	32.0
5....	1 part butter, 2 parts "oleo"	30.8	30.5

From the above it appears that the melting point of a mixture of two or more fats can be readily and accurately calculated from that of its constituents. The agreement, except in No. 1, is within the error of ordinary observation.

Remarks on preceding data.—The mean melting point of the butters examined is 33° 8 C., the maximum is 34° 7 C., and the minimum 32° 6 C. In general terms it may be said that a genuine butter will show a melting point falling within the limits of 33° and 34° C. Of butter adulterants the "neutral lard" has a comparatively high melting point and "oleo-oil" a low one. Unfortunately for analytical purposes it is easy for the fabricator to make an artificial butter whose melting point is sensibly the same as that of the genuine article. On the other hand it is seen that if a false butter be made of a genuine one and only one of the adulterants in common use, the variation of the melting point from the normal will be sufficiently great to call attention to the falsification.

EFFECT OF TIME ON MELTING POINT OF THE FAT DISKS.

By some variations in the melting point of fat disks of different ages my attention was directed to an investigation of the effect of time.

The following data will serve to measure the influence of age on the melting point:

BUTTERS.

Number.	Melting point.		Increase +; decrease—.
	Directly disk was made.	After 24 hours.	
	° C.	° C.	° C.
1.....	33.1	33.6	+0.5
2.....	34.3	34.7	+0.4
3.....	34.2	34.6	+0.4
4.....	34.5	35.2	+0.7
5.....	32.9	32.8	—0.1
6.....	33.1	34.5	+1.4

In every case except No. 5 in the above table it is seen that the melting point of the disks of butter was raised by standing on water at ordinary temperatures for twenty-four hours.

In one instance, a butter whose melting point was 34° 5 C. stood in the form of disks from May 27 until August 3. An attempt was made on this latter date to determine its melting point. At a temperature of 75° C. the disk had not assumed a spherical shape, and the temperature could be carried no higher on account of approaching the boiling point of the alcohol.

ADULTERATED BUTTERS.

Number.	Melting point directly disk was made.	Melting point after—	Increase.
	° C.	° C.	° C.
1.....	34.6	5 days ... 61.4	26.8
2.....	32.9	18 hours .. 37.0	4.1
3.....	38.1	24 hours ... 42.9	5.9
4.....	35.3	46 hours ... 35.3	0.0
5.....	37.8	44 hours ... 40.3	2.5

Again in every case but one a marked rise in the melting point.

"OLEO OIL."

	Melting point.
"Oleo," at once	° C. 29.6
"Oleo," after 42 hours	42.5
Increase.	2.9

It would appear from the above results that adulterated butters and butter adulterants show a greater rise in melting points when the disks

are a day or more old than pure butter. The analytical data, however, are too meager to permit a definite statement of this kind. Should it prove to be true, it would be a valuable indication in the discrimination between pure and adulterated butters. An examination of the old disks with the microscope did not reveal a crystalline structure, and this change, therefore, must be attributed to a molecular modification or superficial oxidation.

EFFECT OF THE PRELIMINARY HEATING OF THE FAT TO DIFFERENT TEMPERATURES.

A butter fat was melted at a low temperature and allowed to stand until the temperature had fallen to $30^{\circ}\text{C}.$; it was still perfectly fluid. The disks were formed by dropping on ice as usual. The melting point obtained was $33^{\circ}\text{C}.$ The fat was now heated to $50^{\circ}\text{C}.$ and treated as above; melting point, $33^{\circ}.4\text{ C}.$ The temperature was then raised to $80^{\circ}\text{C}.$; melting point, $32^{\circ}.8\text{ C}.$

The above results, falling within the possible error of observation, show that the temperature to which the fat is subjected before the formation of the disks has no appreciable effect on the point at which the fat particle becomes a sphere.

EFFECT OF SUDDEN RISE OF TEMPERATURE.

A sudden rise of temperature tends to greatly lower the melting point. A fat which showed a melting point of $35^{\circ}.3\text{ C}.$ when determined in the usual way, melted at once into a perfect sphere when dropped into the water-alcohol mixture having a temperature of $29^{\circ}\text{C}.$ At $28^{\circ}.5\text{ C}.$ the globule was irregular.

A disk of neutral lard, having by the usual method a melting point of $42^{\circ}.4\text{ C}.$, became at once a sphere when dropped into the water-alcohol at $36^{\circ}.2\text{ C}.$ Below that temperature the spheroidal shape was not symmetrical.

In all cases this phenomenon will appear. It may be suggested, therefore, with strict propriety, whether this may not be regarded as the proper melting point. Since the temperature at which the spheroidal state is assumed can be determined within one or two degrees by a preliminary trial, it would not be difficult to have a series of mixtures of water and alcohol arranged so as to show differences of temperature of $0^{\circ}.5\text{ C}.$ By dropping the disks successively into these mixtures the instantaneous fusing point could be determined with accuracy.

The method set forth in the preceding pages has been proved by 165 determinations to be capable of giving agreeing results. Not only will the numbers obtained by the same observer be concordant, but also those of different analysts. This arises from the fact that the moment of the assumption of the spheroidal state is easily determined even by an unpracticed eye. I have also noticed that in this condition pure butter and oleo are quite transparent, while on the other hand neutral

and adulterated butters are still somewhat opalescent. From this it is seen that the data obtained by the old method of determining the temperature of transparency would differ somewhat from those obtained by the proposed procedure. Since the age of the disk has a great deal to do with its melting point, I suggest that all determinations be made within fifteen minutes to two hours from the making of the disks.

The method can also be extended to such bodies as paraffine and bees-wax. The melting point of a paraffine was found to be—

	Melting point.
	° C.
No. 1.....	55.6
No. 2.....	55.1
No. 3.....	55.2
No. 4.....	55.3

An interesting phenomenon was observed in determining the melting point of the paraffine, which may be made to show, in a lecture experiment, the change of volume which bodies sometimes undergo in passing from a solid to a liquid state. The same mixture of water and alcohol used in the examination of fats, allowed the disk of paraffine to sink to about the same point as the disk of fat. When the temperature rose, however, to within one or two degrees of the melting point, there was a sudden increase in volume. The pellet of paraffine rapidly rose to the top of the tube. To avoid this and keep the globule within the liquid I made a mixture of water-alcohol and absolute alcohol. With this arrangement the rise of the paraffine was arrested in the upper third of the tube occupied by the absolute alcohol, where its assumption of the spheroidal state could be readily observed. On placing the tube in a cooling bath the globule of paraffine rapidly sinks as it solidifies. The disks of paraffine and bees-wax are quite irregular, but nevertheless suitable for the process. The melting point of the one sample of bees-wax examined was found to be 64°.2 C.

VISCOSITY.

The speed with which at identical temperatures and pressures different oils flow through an orifice may be used to distinguish them from each other. For a description of the methods used in viscosimetry I refer to Allen's *Com. Organic Analysis*.¹ An ingenious and useful apparatus for viscosimetry has been invented by Babcock.²

Babcock has applied his apparatus to the investigation of the viscosity of butter soaps with promising results.³

¹ Vol. 2, 2d ed., pp. 194 *et seq.*

² Fifth Ann. Rept. Bd. Control N. Y. Exp. Sta., pp. 316 *et seq.*

³ *Ibid.*, pp. 338 *et seq.*

REFRACTIVE INDEX OF OILS.

The use of the refractometer of Abbé in the examination of butters has been proposed by Müller.¹ The principle of the use of this instrument is, that the fats of pure butter possess a less refractive power than the glycerides of a higher molecular weight.

This subject has also been treated by Skälweit.²

ESTIMATION OF SOLUBLE ACIDS IN BUTTER FATS.

*Method of Hehner and Angell.*³—Hehner and Angell, in June, 1874, published a pamphlet on butter analysis in which the details of their method were given.

The following is an abstract of this method:⁴

A weighed quantity, usually 3 grams, of the fat was saponified in a porcelain dish with caustic potash, with frequent stirrings with a glass rod. The clear butter soap was transferred to a flask or retort and decomposed by means of dilute sulphuric acid. This mixture, which contained sulphate of potash, glycerine, and the volatile acids in solution and the insoluble fatty acids floating on the top, was distilled, and the acidity of the distillate estimated by means of a soda solution of known strength. The practical difficulties of this method, such as the violent bumping of the boiling liquid and the impossibility of obtaining a distillate perfectly free from acid, led the authors to adopt a somewhat different method.

This modification is based upon the different percentages of the insoluble fatty acids in butter and other animal fats. The insoluble acids, after saponification, were collected on a moistened filter paper, washed with hot water, and when the soluble acid was washed out, dried and weighed.

They found the percentage of insoluble fats in butter to vary from 85.40 to 86.20, while in other animal fats the percentage of insoluble fatty acids was about 95.5. As will be shown further along, a small error is introduced into this method by washing the insoluble fatty acids on the filter. When this error is avoided, it is found that the percentage of the insoluble fatty acids in butter fat is considerably higher than the figure which has just been given. A detailed description of this part of the process will be given farther on. Turner⁵ suggested the employment of alcohol, with the view to hasten the saponification of the fat; a modification of the process which has been almost universally adopted by analysts.

About 30 or 40cc. of spirits of wine are added to the butter in the porcelain dish and heated over the water bath to near the boiling-point.

¹ *Archiv d. Pharm.*, 1886, p. 210.

² *Rep. d. Ver. Anal., Chem.*, 1886, p. 181.

³ *Analyst*, 1877, p. 147.

⁴ *Hassall, Food and its adulterations*, p. 446.

⁵ *Ibid.*, p. 447.

About 5 grams of solid caustic potash are then added, and from time to time a few drops of water, to facilitate its solution, the liquid being stirred all the time. In this manner the butter becomes rapidly saponified. The clear yellowish solution is then freed from all alcohol over the water bath and the soap decomposed as already described. Care should be taken to remove all the alcohol, as a small quantity of the fatty acids might be held dissolved should any alcohol remain, and so lead to an erroneous result.

*Hehner's method modified by Reichert.*¹—Weigh out $2\frac{1}{2}$ grams of dried and filtered butter fat in an Erlenmeyer flask of 150cc. capacity; add 1 gram of solid potassium hydrate and 20cc. of 80 per cent. alcohol. This mixture is kept upon the water bath with constant shaking until the soap obtained no longer forms a foamy, greasy mass. Afterwards 50cc. of water are added to the flask, and the soap, after it has dissolved in water, is decomposed with 20cc. of dilute sulphuric acid (1cc. of pure sulphuric acid to 10cc. of water). The contents of the flask are now subjected to distillation, with the precaution of conducting through it a slow stream of air, in order to avoid bumping. It is also recommended to use a bulb tube with a wide opening, in order to avoid carrying over the sulphuric acid. The distillate, which, especially with fats poor in butter and by rapid distillation, always deposits a little of the solid fat acids, is filtered through a moistened filter paper and collected in a 50cc. flask. After 10 to 20cc. are passed over it is poured back into the flask and the distillation is now continued until the distillate amounts to exactly 50cc. The distillate, which, when the distillation has gone on evenly, forms a water clear liquid, is immediately titrated with decinormal soda lye after the addition of 4 drops of litmus tincture. The titration is finished if the blue color of the litmus remains constant for some time. Six analyses of an artificial butter fat required 10.5cc. of decinormal soda lye to neutralize the acid in the distillate.

The genuine butter gave on three trials 14.50, 14.45, and 14.60cc., respectively, of the decinormal soda.

Two samples of cocoanut fat required 5.70 and 3.70cc. of soda lye.

Thirteen samples of pure butter required a mean of 13.97cc. of the decinormal soda.

All the other fats which are used in the adulteration of butter required a much smaller amount of the decinormal soda for the saturation of the distilled acid.

In artificial butters the proportion of pure butter and added fat may be calculated from the following formula:

$$B=a(n-b).$$

in which n represents the most probable value of the number representing the quantity of decinormal soda solution required either for pure butter or for the fat with which it may be adulterated. When B equals

¹ Zeit. Anal. Chem., 1879, pp. 68, *et seq.*

O, that is, when the substance contains no pure butter, the value of n may be taken at .30. We have, therefore,

$$O = a(0.30 - b) \text{ from which } b = .30.$$

When B is equal to 100, that is, when the butter is pure, as has already been said, the most probable value of n , according to the thirteen analyses given, is 13.97, or in round numbers $14 \pm .45$, then we have the equation

$$100 = a(14.00 \pm 0.45 - 0.30)$$

and from this the value of

$$a = 7.30 \pm .24.$$

The above equations may therefore be condensed into $B = (7.30 \pm 0.24)(n - 30)$, that is, in order to find the probable butter content of a fat mixture subtract from the number of the cubic centimeters of decinormal soda lye used for titration .30 and multiply the remainder by 7.30. The probable error which will be met with by this estimation amounts to $\pm 0.24(n - 0.30)$.

Medicus and Scherer¹ examined the method of Reichert and found it to be quite exact. For pure butter they found the quantity of decinormal soda lye required should be 13cc.; a mixture of equal parts of butter fat and tallow required 7cc.

Two parts of butter fat and one of tallow required 9.1cc.; three parts of butter fat and one of tallow required 10.1cc.

The authors call attention to the fact that melted butter fat slowly cooled may separate into portions requiring different quantities of the decinormal soda for the saturation of the distilled acid which they afford. Two and one-half to 3 pounds of pure butter fat were used. This was melted and allowed to cool with continued stirring in order to secure a perfectly homogeneous mass. $2\frac{1}{2}$ grams of this mixture, by Reichert's method, required 14cc. of decinormal soda. The fat was now again melted, poured into a large beaker glass, and uncovered allowed to cool without stirring. The solidification took place slowly. After solidification $2\frac{1}{2}$ grams from the upper layer required 13.3cc. of soda.

Allen² also highly recommends Reichert's method. He uses it as follows: Weigh out 25 grams of the clarified butter fat and saponify in a closed flask (a closed flask has been used in the work of the Chemical Division with butter since 1883) with 25cc. of approximately $\frac{N}{2}$ KOH.

Transfer the product to a porcelain basin and evaporate the alcohol at a steam heat. Dissolve the residue in water, add some fragments of pomace coiled round with platinum wire, and distil gently until 50cc. have passed over. Titrate the distillate with $\frac{N}{10}$ caustic alkali using phenol-phthalein as an indicator.

¹Zeit. Anal. Chem., 1880, pp. 159 *et seq.*

²Analyst, 1885, p. 103, *et seq.*

Allen found that a genuine butter fat required not less than $12.5\text{cc.} \frac{N}{10}$ alkali for neutralization of the acid in distillate and that this corresponds to 3.9 per cent. butyric acid distilled over, so that somewhat over 4 per cent. of volatile acids in terms of butyric may be considered to be yielded by the process. (Instead of 3.9 per cent. it should be 4.4 per cent. since $1\text{cc.} \frac{N}{10}$ caustic alkali neutralizes .0088 grams butyric acid.)

Allen gives some comparative results with Reichert's method obtained by different chemists. In the conclusion of the paper Reichert's method is said to be more enlightening than Koettstorfer's for sorting butters.

Modification of Reichert's method by Dr. B. F. Davenport (communicated in MSS.)—Use only 10cc. of alcohol in the saponification; the advantage being that with this small quantity the saponification is almost immediate. In fact I begin to draw out the alcoholic vapor from the flask as soon as it comes to the boil, using a water pump; thus it takes only about fifteen minutes to complete the saponification and the mass evaporated down to a thick mass, free from any alcoholic vapors. My process is to melt the butter at about 80°C. ; filter off the clean fat, stir it into a uniform mass as it solidifies. Weigh off two portions (for I work in duplicate) of the solid mass of 5 grams each upon counterpoised double filters of about the size of the scale pan, using double filters that there may be no chance of anything going through to the scale pan. Roll up the edges of the double filter over the butter upon them, and slip it all into an Erlenmeyer flask. It is easy to get the exact 5 grams upon the open filter, and by putting all into the flask there can be no loss in the transfer. Run off upon the butter in the flask 10cc. of 70 per cent. alcoholic solution, containing 2 grams of KOH. Saponify and get dry mass in about fifteen minutes, add to it 100cc. of water, dissolve, aided by heat, add 50cc. of dilute H_2SO_4 containing about one-tenth part of commercial H_2SO_4 , add several pieces of rough pomace loaded with enough stout platinum wire to lie upon the bottom of the flask, and then distill off 100cc. directly into a sugar flask having a small funnel, and filter in its mouth.

I used at first after distilling off about 20cc. to pour it back into the flask and then distill off 100cc., but I soon learned that that made no difference, as also using a 50cc. sugar flask full of diluted H_2SO_4 , instead of the directed 40cc., which was not quite so convenient a quantity to measure off. I leave the end of the bulb tube connecting the flask with the condenser long enough to enter the condenser so far that there is no need of any rubber connection between them, there being no escaping at the upper end of the condenser of any vapor or of scarcely any odor at all. The operation requires no transferring of the material from the beginning to end. Samples of known pure butter have by this method required an average of 28.8cc. of alkali to neutralize the 100cc.

of distillate. I use phenol-phthalein as an indicator instead of litmus. When the alcoholic KOH solution has been made for some time I make a blank saponification and distillation with that and discount the cubic centimeters of soda solution required by that for that required by the butters.

Reichert's method has also been tried and approved by Caldwell.¹ He says:

For foreign fats Reichert's method was followed with much satisfaction. When all the necessary solutions are once prepared the analysis is made with comparatively little trouble; with less, in fact, than is allowed even by those who praise it most. Its author says that a current of air must be passed through the liquid in the flask while the distillation is going on, to prevent bumping, and Ambuhl says that all attempts to dispense with this precaution by the use of pumice-stone, platinum scraps, and the like failed. Nevertheless, finding it very inconvenient to use the current of air, I ventured to try a combination of short spirals of platinum wire and pieces of pumice stone together, and with complete success; the ebullition continued from beginning to end as quietly as could be desired.

Meissel² has described a modification of Reichert's process as follows:

Five grams of the melted and filtered butter fat are treated in a 200cc. flask with 2 grams of stick alkali and 50cc. 70 per cent. alcohol. After complete saponification the alcohol is evaporated. The soap is dissolved in 100cc. water and decomposed with 40cc. one-tenth H_2SO_4 . The flask is supplied with some pieces of pumice-stone and connected by means of a bulb with a condenser.

The distillation is continued until 110cc. are drawn over. After filtration 100cc. are titrated in presence of litmus with one-tenth N potash and the number of cubic centimeters required increased by one-tenth.

If less than 26cc. of the alkali solution are required in the titration the butter may be suspected of falsification.

Mode of procedure in Reichert's method (used by Dr. C. A. Crampton, Department of Agriculture).—About 2.5 grams of the melted butter fat are weighed out by means of a small pipette and beaker, which are weighed again after the sample has been taken out, and run into a bottle provided with a patent india rubber stopper; 25cc. of a solution of (approximately) semi-normal alcoholic potash is added, the bottle closed and placed in the steam bath until the contents are entirely saponified, facilitating the operation by occasional agitation. The bottle is then removed from the bath, allowed to stand a few moments until partially cooled off, when its contents are transferred to a porcelain evaporating dish, the bottle being rinsed with a little alcohol. The alcohol is then driven off as rapidly as possible, and when the mass of soap and alkali is nearly dry, it is dissolved up in 25cc. of water, and transferred to a suitable flask of about 200cc. capacity, which is fitted with a delivery tube and condenser; the delivery tube is carried up about 8 inches before it is bent to enter the condenser and a bulb is blown in it just below

¹ Second Ann. Rept. N. Y. S. Bd. of Health, p. 526.

² Ding. Poly. J., vol. 233, p. 229.

the elbow and filled with broken glass or glass wool. After the soap solution has been transferred to this flask, the evaporating dish is rinsed out with 25cc. more water, which is added to the contents of the flask, and the fatty acids are then set free by the addition of 20cc. of a solution of phosphoric acid,¹ making the liquid measure in all about 70cc. Heat is applied gently at first, and gradually increased until the distillate comes over regularly. When 50cc. have distilled off the operation is finished and the distillate is titrated with one-tenth alkali, using phenolphthalein as an indicator.

I have adopted phosphoric acid in preference to sulphuric for setting free the fatty acids, because it is not so liable to carry over as the latter; much greater care is necessary when sulphuric acid is used. Before the modification of the delivery tube was adopted, I frequently found H_2SO_4 in the distillate. Thus, before using the bulb, two blank experiments required 1.8 to 2.0cc., one-tenth alkali, for neutralization and gave a perceptible precipitate of $BaSO_4$. After adding the bulb I found blanks occasionally to require as much as .8cc. when the distillation had not been carefully watched. The following comparative results show that there is practically no difference which acid is used, when the operation is carried on with care. The processes used were identical, except that in the second, 20cc. of 10 per cent. sulphuric acid was substituted for the phosphoric acid. The results are for 2.5 grams of fat.

No. 1	{ With phosphoric acid	12.7	12.6	12.7		
	{ With sulphuric acid	12.7	12.7	12.6		
No. 2	{ With phosphoric acid	15.8				
	{ With sulphuric acid	15.3				
No. 3	{ With phosphoric acid	13.1				
	{ With sulphuric acid	13.2				
No. 4	{ With phosphoric acid	15.3	14.1	14.5		
	{ With sulphuric acid	14.8	14.6	14.9	14.5	15.0

Blanks should always be run, and will be generally found to require .1 to .3cc. of the deci-normal soda before they will show the color with the phenol indicator.

*Koettstorfer's process*² (as used in this laboratory).—About 2.5 grams butter fat (filtered and free from water) are weighed into a patent rubber-stoppered bottle and 2½cc. (approximately) semi-normal alcoholic potash added. The exact amount taken is determined by weighing a small pipette with the beaker of fat, running the fat into the bottle from the pipette and weighing beaker and pipette again. The alcoholic potash is measured always in the same pipette and uniformity further insured by always allowing it to drain the same length of time (thirty seconds). The bottle is then placed in the steam bath together with a blank, containing no fat. After saponification is complete, and the bottles cooled down, the contents are titrated with accurately semi-normal hydro-

¹ Made by dissolving 200 grams of commercial glacial phosphoric acid in a litre of water; its specific gravity is 1.140.

² Zeit. Anal. Chem. 1879, p. 199; Analyst, 1879, p. 106.

chloric acid, using phenol-phthalein as an indicator. The number of cubic centimeters of the acid used for the sample deducted from the number required for the blank gives the number of cubic centimeters which combines with the fat, and the saturation equivalent is calculated by the following formula, in which W equals the weight of fat taken in milligrams and N the number of cubic centimeters which has combined with the fat.

$$\text{Sat. Equiv.} = \frac{2 W}{N}.$$

For pure butters the mean value of N is about 17 when 2.5 grams of butter fat are taken, and the saturation equivalent may vary from 230 to 255. On the other hand for lards, tallows, and other fats commonly used for adulterants the equivalent rises to 270 and 290. These numbers, therefore, give a fair idea of the purity of a butter, or if an adulteration has been practiced, of its extent.

ESTIMATION OF INSOLUBLE ACIDS IN BUTTER FAT.

*Method of Hehner.*¹—This method consists in saponifying the fat with alcoholic caustic potash, subsequent evaporation of the alcohol, decomposition of the soap with sulphuric or hydrochloric acid, and the determination of the insoluble acid gravimetrically.

The process as originally described by Hehner is carried on as follows:

The filtered butter fat is weighed in a beaker glass with a glass rod; 3 or 4 grams are taken out by means of the glass rod and put in an evaporating dish about 5 inches in diameter; the glass rod with the fat which remains on it is left in the evaporating dish. The beaker glass is again weighed and the amount of butter fat determined from the difference in weight. To the weighed fat are added 50cc. alcohol and 1 to 2 grams of pure caustic potash. The alcohol is warmed gradually upon the water bath, by which the butter fat, especially when stirred with the glass rod, easily dissolves to a clear yellow liquid, giving off a distinct odor of butyric ether. The heating is continued for about five minutes and distilled water is then added drop by drop to the mass. If this produces a cloudiness in the liquid, due to the separation of undecomposed fat, the heating is continued somewhat longer until finally the further addition of water does not produce the least cloudiness. Should, however, through the careless addition of water, some fat separate in the form of oily drops which do not again easily pass into the solution in the diluted alcohol, the whole mass must be evaporated to dryness and treated anew with alcohol, or the experiment be done over again with some fresh fat.

The clear soap solution is now evaporated on the water bath to the consistency of sirup in order to remove the alcohol, and the residue

¹ Zeit. Anal. Chem. 1877, pp. 145 et seq.

dissolved in 100 to 150cc. of water. To the clear liquid hydrochloric or sulphuric acid is added to a strongly acid reaction, in order to decompose the soap. The insoluble fat acids are now separated out as a cheesy mass, which for the most part quickly rise to the surface. The heating is continued for a half hour until the fat acids are melted to a clear oil and the acid aqueous liquid is almost completely clear. Meanwhile a thick Swedish filter paper of 4 or 5 inches in diameter has been dried in a water bath. The filter paper must be of the best quality and so thick that even hot water will only pass through it drop by drop. A small beaker glass is now weighed, afterwards a filter tube, and then the filter tube and the filter; in this way is obtained the weight of the filter and the beaker glass.

The weighed filter is now fitted to a funnel moistened and half filled with water. The aqueous liquid and the melted fat are then poured out of the evaporating-dish into the filter, and the dish and glass rod are washed with boiling water. There is no difficulty in bringing all of the fat on the filter, so that the evaporating dish does not appear in the least greasy. To make sure, however, the dish can be washed with ether and the liquid obtained added to the fatty acids.

The fatty acids are washed upon the filter with boiling water. The filter should be never more than two-thirds full. If the filtrate tested with sensitive litmus tincture does not appear acid, the rest of the water is allowed to run through, and the funnel is dipped into a beaker-glass filled with cold water, so that the surface of the liquids within and without the funnel are at the same level. As soon as the fatty acids have solidified the filter is taken out of the funnel, placed in the weighed beaker-glass, and dried in a water-bath to constant weight. The drying is continued for two hours and the filter paper is then weighed. It is again dried for two and a half hours and weighed a second time. It must be remembered that it is not a mineral substance which is under treatment, but an easily-oxidizable fat, so that an exact constant weight cannot be expected.

Butter fat gives between $86\frac{1}{2}$ and $87\frac{1}{2}$ per cent. of insoluble fatty acids, though in some cases the number may rise to 88 per cent. On the other hand, the animal fats give about $95\frac{1}{2}$ per cent. of insoluble fats.

It must be expected that the kind of food which cows receive influences considerably these numbers. In order to determine this important point, Dr. Turner had a cow fed for a long time exclusively on oil cake, with the object of raising the percentage of insoluble fat acids to the highest point. It is worthy of remark, however, that the butter so produced gave the unusually low percentage of 86.3.

*Method of Muter.*¹—The total fatty acids. About 10 grams (or 150 grains) of the butter fat at 100° F. are weighed by difference from a suspended tube into a clean, dry 15-ounce flask, and 5 grams of potassium hydrate with 2 fluid ounces of rectified spirit are added. The flask is

¹ Analyst, 1877, pp. 10, 11.

placed in a basin with hot water, and kept boiling for a considerable time, until on adding water not the faintest turbidity occurs. Ten ounces of water are added, the evaporation continued (just short of boiling) until all traces of alcohol are dissipated. The contents of the flask are then made up to 7 ounces with nearly boiling water, and a good-fitting cork having been introduced through which just passes a tube 2 feet long and ending in a small funnel, 5 grams of full strength sulphuric acid are poured in down the tube followed by some water. The whole is then agitated with a circular motion until the soap, which rises suddenly, is changed into a perfectly clear and transparent stratum of fatty acids. The flask and contents are then cooled down to 40° F., till a perfectly solid cake of fatty acid forms. A few drops of cold water are run in to wash the tube, and, the cork having been removed, a small piece of fine cambric is placed over the mouth of the flask, held *in situ* by an ordinary India-rubber ring. The fat cake is caused to detach itself from the sides of the flask by a gentle movement, and then the filtrate is decanted, without breaking the cake, into a litre test mixer with a good stopper. About an ounce of cold water is poured into the flask through the cambric, and the whole cake and flask rinsed out by gently turning round, and the washings added to the filtrate. Six ounces of water at 120° F. are now added through the muslin, which is then quickly detached, and the cork and tube inserted; the whole again heated, this time to 200° F., and kept constantly agitated with a circular but not a jerky motion for five minutes. This agitation so divides the fat that it almost forms an emulsion with the water, and is the only means of thoroughly and rapidly washing fatty acids without loss. In practice no butyric acid comes off at 200° F., but any trace that might do so is caught in the long tube. The cooling and filtering are then again proceeded with as above described (the filtrate being added to the contents of the test mixer), and the washings are repeated alternately, cold with 1 ounce, and hot with 6 ounces of water, until they do not give the slightest change to neutral litmus. After thoroughly draining the residual cake by letting the flasks stand upside down for some time, the cambric is removed and the flask is laid on its side in the drying oven, with a support under the neck, until the acids are thoroughly fused, when they are poured while hot into a tarred platinum capsule, dried and weighed. The film of fatty acid still remaining on the flask is rinsed out with ether and dried in a small weighed beaker, and the weight added to the whole. If any drops of water be observed under the fatty acids in the capsule after an hour's drying the addition of a few drops of absolute alcohol will quickly cause them to dry off. If any trace of fat is on the cambric it should be also dried and extracted with ether, but with care not to break the cake at the last pouring off this does not occur.

The process is absolutely accurate, and the merest tyro cannot make any loss so long as he does not deliberately shake the melted acids

against the cork, which he could not do if he practiced a circular agitation while washing.

The filtrate in the test mixer is now made to a definite bulk of 1 litre, and in 200cc. the total acidity is taken with a weak solution of sodium hydrate. The solution I generally use represents .01 of NH_3 in each cubic centimeter, as it serves also for nitrogen combustions; but a useful strength would be decinormal soda, containing .004 NaOH in each cubic centimeter. The acidity found is multiplied by 5, calculated to H_2SO_4 and noted as "total acidity as H_2SO_4 "; 100cc. are next taken, and precipitated with barium chloride in the presence of a strong acidulation, with hydrochloric acid, well boiled and washed by three decantations, boiling each time; and lastly on a filter, till every trace of soluble barium is removed. The precipitate is dried, ignited, and weighed as usual, multiplied by 10, and calculated to H_2SO_4 and noted as "total sulphuric acid." Lastly, 100cc. are evaporated to dryness over the water bath in a tarred platinum dish holding 120cc. and furnished with a cover of platinum foil, also tarred. When dry the dish is covered and heated over a Bunsen till all fumes cease, and, a fragment of pure ammonium carbonate having been added, the whole is again ignited and weighed. The amount of potassium sulphate found is multiplied by 10 and calculated to H_2SO_4 and noted as "combined sulphuric acid."

OTHER METHODS.

Liebschütz¹ has described a method for the examination of butter and oleomargarine, being a modification of David's process.²

The fatty acids are saponified by baryta in alcoholic solution. The alcohol is evaporated and the glycerine washed out. The excess of baryta is removed by exactly neutralizing with sulphuric acid and filtering. The residue, however, is not merely a mixture of glycerine and water. The addition of alcohol in excess throws down a considerable quantity of salts which have remained in solution. The alcohol is again evaporated and the glycerine obtained, dried, and weighed. Pure butter yields about 13.7 per cent. of glycerine in this way, while oleomargarine yields only 7 per cent. The glycerine from butter when ignited left about 5 per cent. ash (barium) while that from oleo left only .3 to .6 per cent.

RESULTS OF HANSSEN'S INVESTIGATIONS.

Dr. August Hanssen³ has made a comparative study of the more important methods of analysis mentioned in the foregoing pages and has reached the following conclusions:

- (1) The determination of the melting-points of the different fats is to be strongly recommended.

¹ Analyst, 1885, p. 111, *et seq.*

² Compt. Rend., 1886, vol. XCIV, p. 1427.

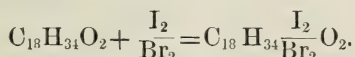
³ Studien über den chemischen Nachweis fremder Fette im Butterfette, p. 34.

- (2) The elementary analysis of the fats gives no indication whether adulteration has been practiced or not.
- (3) Butter fat is not easily decomposed by heat. With a rise of temperature the decomposition is at first, for the greater part, confined to the glycerides of the non-volatile acids.
- (4) In the saponification of butter fat by Hohner's method there is no appreciable loss of ethers. There is also no loss of volatile acids in direct saponification in alcohol.
- (5) For the detection of foreign fats in butter, the best method is that of Reichert-Meissel, and next that of Koettstorfer.
- (6) For a comparative test of the various methods the mean for insoluble acid (Hohner) is taken at 87.50 per cent. ; for Koettstorfer's equivalent 227, and for Reichert-Meissel 28.8.
- (7) The washing out of the soluble acids must not be carried too far ; for 2 to 2.5 grams of fat three litres of water seem best.

ABSORPTION OF BROMINE AND IODINE BY BUTTER FATS.

Oleic acid is capable of absorbing for each formula molecule one molecule of bromine or iodine. Stearic acid does not possess this property. Therefore it is easy to approximately determine the relative quantities of these two acids when present in the same fat by the quantity of the halogen absorbed.

Thus (stearic acid) $C_{18}H_{36}O_2$ does not absorb bromine and iodine, while (oleic acid) does.



The glycerides of the above acids, *i. e.*, the natural fats, have the same absorptive power as the acids themselves.

Mills, Snodgrass & Akitt¹ have determined the quantities of bromine absorbed by various fixed oils. The method employed is as follows :

The weight of dry oil taken is about .1 gram ; this is dissolved in a stoppered bottle of 100cc. volume by 50cc. dry CCl_4 . To this is now added a solution of about 8 grams per litre of bromine dissolved in CCl_4 . The addition of this reagent is continued until a permanent coloration is produced at the end of fifteen minutes.

If greater accuracy is required an excess of bromine may be added, afterwards treated with a solution of KI and some starch, and titrated with a standard solution of sodium thiosulphate.

The excess of bromine may also be determined by titration with a standard solution of β -naphthol in CCl_4 .

Hübl² has described the reactions of fats with iodine.

The reagents employed are an alcoholic solution of iodine and $HgCl_2$, in the proportion $I_2 : HgCl_2$.

The iodine is dissolved (25 grams) in absolute alcohol (500cc.) The mercuric chloride is also dissolved (30 grams) in nearly absolute alcohol

¹ Journ. Soc. Chem. Industry, vol. 2, p. 435, and vol. 3, p. 366.

² Ding. Poly. J., vol. 243, p. 281.

(500cc.). After filtering it is added to the solution of iodine. After standing twelve hours its iodine strength is determined by titration with decinormal solutions of sodium thiosulphate. From .8 to 1 gram of the fat is dissolved in 10cc. chloroform. To this, in a stoppered bottle, is added the solution of iodo-mercuric chloride (20 to 30cc.) After standing for two hours the solution must still be brown.

Add now 10 to 15cc. 10 per cent. water solution of KI and dilute with water to 150cc. The free iodine is then determined by standard thiosulphate of sodium. The compound formed when pure oleic acid is treated as above is chloro-iodo-oleic acid ($C_{18}H_{34}IClO_2$).

Moore¹ has tried Hübl's method and finds it valuable.

The fat of butter containing less oleic-glycerides than the fats ordinarily used as adulterants for butter shows, consequently, less bromine or iodine absorption :

Kind of fat.	Absorption of bromine.	Absorption of iodine.
	<i>Per cent.</i>	<i>Per cent.</i>
Butter fat	24.5 to 27.9	26. to 35.
Lard	37.3	59. 61.9
Tallow	-----	40.
Cotton-seed oil	50.	105. 109.
Cocoanut oil	5.7	8.9

The method is therefore of value in determining the nature of the fat under examination.

If there be a mixture of two fats the methods will also give a fairly good approximation of the percentages of each.

Thus, let x be the percentage of one fat and y of the other.

Then—

$$x + y = 100$$

Let m be the representative of the iodine absorption of x and n of y , and let Λ be the number found for the mixture.

Then—

$$x = \frac{100 (\Lambda - n)}{m - n}$$

Jones² points out the changes which butter fats undergo when kept for a long while at a high temperature. He notices in a few hours that the specific gravity of such a fat kept at 100° F. increased from 912.1 to 912.6. He uses the following method of estimating the insoluble fatty acids :

REAGENTS.

- Twenty-eight grams roughly weighed of the best potassium hydrate dissolved to a litre with alcohol, specific gravity .840.
- Twenty-five grams of strong sulphuric acid made up to a litre of distilled water.
- Decinormal soda solution of exact strength.

Saponification is carried on in flasks about 250cc. capacity. About 5 grams of butter fat are used for each saponification. The alcoholic

¹ Am. Chem. Jour., vol. 6, p. 416.

² Analyst, 1878, pp. 19 *et seq.*

potash is measured by 50cc. pipette, which is allowed to drain into each flask for exactly the same length of time. The flasks are closed with glass marbles, placed upon the water bath and saponified at a temperature of about 50° C. After perfect solution has taken place they are allowed to remain for an hour or two and then diluted with slightly warmed distilled water. Into each flask and likewise into two beakers containing 50cc. of the alcoholic potash are now run about 1cc. of the approximately semi-normal acid more than is necessary to neutralize the 50cc. of alcoholic potash. The excess of the acid over the potash is afterwards determined by the decinormal soda. The flasks after the addition of the acid are nearly filled with water and gently agitated, then placed on the water bath until the fatty acids form a clear stratum. They are then allowed to cool and stand over night. On the following morning the solutions from the cakes of fat are poured into a filter. When the whole solution is on the filter the flasks are rinsed with 15 to 20cc. of cold distilled water, and when this is poured off about 150cc. of hot water are added and the flasks briskly shaken for a minute or two. Two good washings with hot water are believed to be enough. The filtrates are now treated with the decinormal soda, the amount for the excess of sulphuric acid deducted, the remainder being the index of the soluble acids of the butter, which are calculated as butyric acid.

The insoluble fatty acids in the flasks and the small amount that may have passed on to the filter paper are allowed to remain until the following day, by which time the latter become air-dried and in a fit state to rinse with ether. The fat in the flasks is then melted and poured, together with the rinsings of the ether, into counterpoised dishes with perpendicular sides, about 3 inches across and 1½ inches deep, and the filter papers are also thoroughly washed with ether, the funnels being covered during the process. After the evaporation of the ether a little absolute alcohol is added, the dishes dried in the water-bath for half an hour, cooled, and weighed. Afterwards they are again dried for twenty minutes and reweighed.

For a more convenient method of manipulating fatty acids, Blyth¹ has recommended the following:

The flask in which the saponification is made should be of 300 to 400cc. capacity, with a rather long and narrow neck, furnished with an accurately fitting stopper, through which two tubes pass, one provided with a stop-cock to let out the liquid, and therefore terminating on a level with the interior surface of the stopper, the other to let in the air, prolonged to nearly the bottom of the flask and externally bent siphon-like. The fat is saponified in the flask and the soap decomposed in the usual way; when this is effected, the stopper is inserted, and the flask is turned upside down and kept in that position during the entire washing process. Directly the whole of the fat has risen to the surface the lower liquid is run off, whilst hot or cold water is introduced by opening

¹ Analyst, 1878, p. 112.

the stopper under the water and simultaneously sucking at the syphon. Thus all waiting for the fat to cool is discarded, and reasonable quantity of water can be rapidly used to thoroughly wash the fatty acids, and a filter is not required.

DETERMINATION OF SOLUBLE AND INSOLUBLE FAT ACIDS.

METHOD ADOPTED BY ALLEN.¹

(a). Dissolve 14 grams of good stick-potash in 500cc. of rectified spirit, or methylated spirit which has been redistilled with caustic alkali, and allow the liquid to stand till clear. This solution will be approximately seminormal.

(b). A standard hydrochloric or sulphuric acid of approximately seminormal strength.

(c). Accurately prepared decinormal caustic soda. Each 1.0cc. contains .0040 grams of NaOH and neutralizes, .0088 grams of butyric acid, $C_4H_8O_2$.

A quantity of the butter fat (separated from water, curd, and salt, as described on page 152) is melted in a small beaker, a small glass rod introduced, and the whole allowed to cool, and then weighed. It is remelted, stirred thoroughly, and about 5 grams poured into a strong 6-ounce bottle. The exact weight of fat taken is ascertained by reweighing the beaker containing the residual fat.

By means of a fast-delivering pipette 50cc. measure of the alcoholic potash (solution a), is run into the bottle, and the pipette drained exactly thirty seconds. At the same time another quantity of 50cc. is measured off in an exactly similar manner into an empty flask.

The bottle is fitted with an india-rubber stopper, which is tightly wired down, and is placed in the water-oven, and from time to time removed and agitated, avoiding contact between the liquid and the stopper. In about half an hour the liquid will appear perfectly homogeneous, and when this is the case the saponification is complete, and the bottle may be removed. When sufficiently cool, the stopper is removed, and the contents of the bottle rinsed with boiling water into a flask of about 250cc. capacity, which is placed over a steam bath, together with the flask containing merely alcoholic potash, until the alcohol has evaporated.

Into each of the two flasks is now run about 1cc. more seminormal acid (solution b) than is required to neutralize the potash, and the quantity used accurately noted. The flask containing the decomposed butter fat is nearly filled with boiling water, a cork with a long upright tube fitted to it, and the whole allowed to stand on the water-bath until the separated fatty acids form a clear stratum on the surface of the liquid. When this occurs the flask and contents are allowed to become perfectly cold.

Meanwhile the blank experiment is completed by carefully titrating the contents of the flask with the decinormal soda, a few drops of an alcoholic solution of phenolphthalein being added to indicate the point of neutrality.

The fatty acids having quite solidified, the resultant cake is detached by gently agitating the flask, so as to allow the liquid to be poured out, but avoiding fracture of the cake. The liquid is passed through a filter to catch any flakes of fatty acids, and is collected in a capacious flask. If any genuine butter be contained in the sample, the filtrate will have a marked odor of butyric acid, especially on warming.

Boiling water is next poured into the flask containing the fatty acids, a cork and long glass tube attached, and the liquid cautiously heated till it begins to boil, when the flask is removed and strongly agitated till the melted fatty acids form a sort of emulsion with the water. When the fatty acids have again separated as an oily layer, the contents of the flask should be thoroughly cooled, the cake of fatty acids detached,

¹ Commercial Organic Analysis, vol. 2, 2d ed., pp. 156 *et seq.*

and the liquid filtered as before. This process of alternate washings in the flask by agitation with boiling water, followed by cooling, and filtration of the wash-water, is repeated three times, the washings being added to the first filtrate. It is often difficult or impossible to obtain the wash-water wholly free from acid reaction, but when the operation is judged to be complete the washings may be collected separately and titrated with decinormal soda. If the measure of this solution required for neutralization does not exceed 0.2cc. further washing of the fatty acids is unnecessary.

The mixed washings and filtrate are next made up to 1,000cc., or some other definite measure, and an aliquot part carefully titrated with decinormal soda (solution c). The volume required is calculated to the whole liquid. The number so obtained represents the measure of decinormal soda neutralized by the soluble fatty acids of the butter fat taken, plus that corresponding to the excess of standard acid used. This last will have been previously ascertained by the blank experiment. The amount of soda employed in this is deducted from the total amount required by the butter fat quantity, when the difference is the number of cubic centimeters of standard soda corresponding to the soluble fatty acids. This volume multiplied by the factor 0.0088 gives the butyric acid in the weight of butter fat employed.¹

The flask containing the cake of insoluble fatty acids is thoroughly drained and then placed on the water-bath to melt the contents, which are poured as completely as possible into the (wet) filter, through which the aqueous liquid was previously passed. The fatty acids are then washed on the filter with boiling water, to remove the last traces of sparingly soluble acids. The filter is then placed in a small dry beaker and treated in the manner described on page 38, the main quantity of fatty acids and the supplementary portion subsequently dissolved out of the flask and filter being weighed separately.²

When it is only required to determine the insoluble acids of butter fat the foregoing tedious mode of operating may be avoided by diluting the soap solution obtained by saponifying 5 grams of the fat till it measures about 300cc. The large excess of alkali is then neutralized by cautious addition of hydrochloric acid, and the hot solution treated with a slight excess of barium chloride or magnesium sulphate. The precipitated barium or magnesium soap is well washed with hot water, and then rinsed off the filter into a separator, where it is decomposed by dilute hydrochloric acid. The resultant layer of insoluble fatty acids is washed by agitation several times with warm water, and is then treated as directed on page 38.

In the analysis of butter fat, the sum of the insoluble fatty acids by weight and of the soluble fatty acids, calculated as butyric acid, should always amount to fully 94 per cent. of the fat taken. In the author's own experience the sum more frequently approaches or even exceeds 95 per cent., especially if the butter be adulterated.

The soluble fatty acids, calculated as butyric acid, should amount to at least 5 per cent., any notably smaller proportion being due to adulteration.³ The insoluble fatty

¹ Thus, suppose an experiment to have given the following figures: Weight of butter fat taken, 5.120 grams; decinormal soda required in the blank experiment, 3.90cc.; decinormal soda required to neutralize one-fifth of the solution of the soluble fatty acids, 6.25cc.; then

$$\frac{.008 (31.25 - 3.9) \times 100}{5.120} = 4.70 \text{ per cent.}$$

² Instead of weighing the insoluble fatty acids, W. F. Perkins has proposed to dissolve them in alcohol, and titrate with standard alkali in the manner described on page 76. The objection to this plan is the somewhat variable character of the fatty acids themselves. Calculating their neutralizing power on the assumption that they are wholly stearic acid, Perkins found 92.0 and 91.7 per cent. of insoluble acids in pure butter fat. Calculated to oleic acid these figures would not be materially modified, but their equivalents in palmitic acid are 83.3 and 83.0 per cent. respectively.

³ According to J. Bell, the proportion of soluble acids calculated as butyric acid not unfrequently falls as low as 4.5, and the percentage of insoluble acids sometimes slightly exceeds 89.0.

acids from genuine butter fat rarely exceed $83\frac{1}{2}$ per cent., occasionally reaching 89 per cent., but a sample ought scarcely to be regarded as certainly adulterated unless the insoluble acids exceed $89\frac{1}{2}$ per cent. As a standard for calculation 88 per cent. of insoluble acids¹ may be regarded as a fair average, the soluble acids being taken at $5\frac{1}{2}$ per cent.

Allen, in a later contribution to the literature of Reichert's method, says:²

A further experience in the employment of Reichert's process for examining fats has led me to abandon the expression of the results in terms of butyric acid, in favor of a statement of the weight of caustic potash neutralized by the distillation from 100 grams of the oil. This is obtainable by multiplying the volume of decinormal alkali neutralized by the distillate from 2.5 grams by the factor 0.2244.³

The following table contains a number of results expressed in both ways:

	C. C. of $\frac{N}{10}$ alkali re- quired by 2.5 grams.	KOH re- quired by 100 parts of oil.	Observer.
Butter or milk fat;			
Cow's	12.5 to 15.2	2.80 to 3.41	Reichert, Caldwell, Moore, Allen, &c.
Ewe's	13.7	3.07	Schmitt.
Goat's	13.6	3.05	Do.
Porpoise's	11.3	2.51	Allen.
Cocoanut oil	3.5 to 3.7	0.78 to 0.83	Reichert, Moore, Allen.
Palm-nut oil	2.4	0.54	Allen.
Balm oil	0.8	0.18	Moore.
Cacao butter	1.6	0.36	Do.
Butterine and oleomargarine	0.2 to 1.6	0.04 to 0.36	Caldwell, Moore, Allen.
Whale oil	3.7	0.83	Allen.
Do	12.5	2.80	Do.
Porpoise oil	11.0 to 12.0	2.47 to 2.69	Do.
Sperm oil	1.3	0.29	Do.
Bottle-nose oil	1.4	0.31	Do.
Menhaden oil	1.2	0.27	Do.
Cod-liver oil	1.1 to 2.1	0.24 to 0.47	Do.
Sesame oil	2.2	0.48	Do.
Cotton-seed oil	0.3	0.07	Moore.
Castor oil	1.4	0.31	Allen.

From these results it is evident that the fats of different kinds of milk (butter fats) are sharply distinguished from nearly all other fats by the large proportion of soluble volatile fatty acids they yield by Reichert's process. The most remarkable exception is presented by porpoise oil and some samples of whale oil. In porpoise oil I have found 5 per cent. of valeric acid, and Chevreul obtained as much as 9.63 per cent. In a recent paper I pointed out that in porpoise butter the glyceride of valeric acid appeared to replace the butyryn characteristic of the butter of terrestrial mammals.

Some of the chemists who have employed Reichert's process take the precaution to filter the distillate before titrating it, so as to get rid of any volatile acids which may be insoluble or very sparingly soluble in water. This plan may sometimes be adopted with great advantage. Thus when the solution of the soap obtained by saponifying cocoanut or palm-nut oil is acidulated and distilled, a notable proportion of lauric acid passes over and solidifies in the condenser or on the surface of the distillate; and

¹The percentage of adulterant in a butter fat may be calculated from the following formula, in which F. is the percentage of foreign fat and I that of the insoluble fatty acids: $F = (1 - 88) \times 13.3$. Or each 0.1 per cent. of soluble acids above 0.5 may be regarded as showing the presence of 2 per cent. of butter fat.

²Analyst, 1887, pp. 11 et seq.

³1 cc. of $\frac{N}{10}$ alkali contains 0.00561 gram of KOH; and $\frac{.00561 \times 100}{2.5} = .2244$

by adding water to the contents of the retort, again distilling, and repeating this process several times, a very considerable proportion of volatile fatty acids can be obtained from cocoanut oil. In assaying butter, the appearance of insoluble acids in the distillate would furnish a valuable indication of the presence of cocoanut oil, and they should be removed by filtration, or the distillate will be found to neutralize so large a volume of alkali as considerably to diminish the practical value of the process as a means of distinguishing butter from butter substitutes, as has been pointed out by Moore and others. Latterly, I have adopted the plan of filtering the distillate in all cases, washing the filter with cold water, and then immersing the filter, with any adhering insoluble acids, in alcohol, which is then titrated with decinormal alkali and phenol-phthalein. In the case of ordinary butters and butter substitutes the insoluble volatile acids only neutralize about 0.2cc. of decinormal alkali.

The question having recently been raised, the following experiments were made at my request by Mr. William Barraclough on a sample of butter fat, in order to ascertain the variation in the results of Reichert's process produced by modifications in the methods of conducting the saponification and distillation:

(1) Two and a half cubic centimeters of butter fat was saponified by alcoholic potash in an open basin, the alcohol evaporated off completely at a steam heat, the residual soap dissolved in water, the solution acidulated with sulphuric acid in slight excess, diluted to 75cc. and distilled gently in a globular flask with side tubulure adapted to a condenser until 50cc. had passed over. The flask held 460cc. up to the side tube, and some fragments of pumice-stone coiled round with platinum wire were added to the contents to promote evolution of vapor.

(2) An exact repetition of No. 1 experiment.

(3) Saponification was effected in a flask furnished with a long tube and heated by steam. The subsequent manipulations were the same as in experiment 1.

(4) Saponification was effected in a well-closed bottle placed in the water oven. Other manipulations unchanged.

(5) Manipulation exactly as in experiment 3, except that the distillation was conducted in a flask fitted to the condenser by a cork and bent tube.

(6) Conducted as in experiment 3, except that the distillation was conducted in a retort.

(7) Blank experiment with the alcoholic potash employed in the previous experiments, the manipulation being that in experiment 3. The alcoholic potash was brown and not very recently prepared.

Experiments.

	Decinormal alkali for 2.5 grams.
	cc.
No. 1.....	11.80
No. 2.....	11.85
No. 3.....	12.40
No. 4.....	12.50
No. 5.....	12.40
No. 6.....	12.45
No. 7.....	0.25

These results show that a sensible loss occurs if the saponification be conducted in an open basin, doubtless owing to the formation of butyric ether. On the other hand, the exact nature of the distilling apparatus appears to be of little importance. This latter conclusion is not in accordance with the experience of some other chemists.

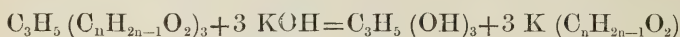
Zulkowsky and Groger¹ have studied and modified Haussman's method² of volumetric fat analysis. This method is based on the fact

¹Ber. Chem. Gesel., vol. 16, p. 1140.

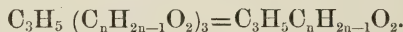
²Ding. Poly., J., vol. 244, p. 303, and vol. 246, p. 286.

that an alcoholic solution of a fat acid is immediately saponified by the addition of alcoholic potash, while a neutral fat requires time and heat to secure complete saponification.

When, therefore, an alcoholic solution of fat acids and neutral fats to which phenol-phthalein has been added is titrated with caustic potash, the red color only appears when the fat acids are saponified, and only comes permanently when all the fats are saponified. When the red color appears an excess of caustic potash is added and the whole boiled for half an hour to saponify all the neutral fats and retitrated, whereby the amount of caustic potash required to effect the saponification of all the fats is ascertained, and the quantity of potash required for each titration represents the relative proportion of fat acids and neutral fats in the mixture operated on. When a neutral fat is saponified the following reaction takes place:



and therefore every litre of normal potash splits up one-third equivalent of glycerine, *i. e.*, 30.667 grams. One cubic centimeter normal potash is therefore equivalent to 0.030667 gram glycerine. The theoretical yield of fat acids could also be calculated by the following formula:



Then one litre normal potash represents one-third equivalent of glycerine residue, or 12.667 grams. If 5cc. normal potash have been employed the weight of the glycerine residue would be $.012667 \times 5$.

F. W. A. Woll¹ gives the results of his studies with butter and artificial butter.

Mixtures of pure butter with "oleo oil" were made and examined by the methods of Koettstorfer and Reichert, and the results compared with theory. The following numbers were obtained:

Per cent. butter.	Koettstorfer.			Reichert.		
	Calculated.	Found.	Difference.	Calculated.	Found.	Difference.
	<i>mg.</i>	<i>mg.</i>	<i>mg.</i>	<i>cc.</i>		<i>cc.</i>
20.....	200.8	201.4	+0.6	2.98	3.11	+0.13
40.....	206.2	207.3	+1.1	5.81	6.39	+0.58
50.....	208.5	209.0	+0.5	6.55	7.08	+0.53
60.....	211.5	212.7	+1.2	8.65	9.06	+0.41
80.....	217.7	215.6	-2.1	10.37	11.56	+1.19
Means			1.1			0.57

NOTE.—It is very easy to get exact results by the above method of mixture. Butter and an oil are used whose behavior with the reagents employed was determined by preliminary experiment. The case is very different when the analyst is called on to examine an *unknown* sample. The butter in an unknown sample may have quite a different per cent. of volatile acid from that used in the samples given. The value of this method, therefore, is seriously impaired for determining the extent of adulterations in case where the separate examination of the constituents is impossible.

¹Zeit. Anal. Chem., 1884, p. 28, and Am. Chem. Jour., vol. 9, p. 62.

The author gives a table of the analyses of 37 samples of butter and butter substitutes giving the percentage of water, the specific gravity at 37° C., the melting point determined by the method of Blyth, the milligrams of KOH required in Koettstorfer's method and of cubic centimeters by Reichert's method.¹

The author concludes that the melting point is of no value in discriminating between pure and false butters, but the specific gravity, the saponification process, and the distillation of the volatile acid are sufficient to distinguish at once between the true and the false.

The oleo oil employed had a mean specific gravity at 37° C. of 0.90369 and its melting point was 27° C. The "neutral" had a specific gravity of 0.9053 and a melting point of 38° C.

BEHAVIOR OF COCOANUT OIL WITH SOME OF THE METHODS USED IN ANALYSIS OF BUTTER FATS.

R. W. Moore, in a paper read before the American Chemical Society, September 18, 1885,² calls attention to cocoanut oil as a substitute for butter. He gives its fusing point at 24° C. to 24° C., and calls attention to the fact that its specific gravity is higher than that of butter fat. It is also noticed that the insoluble acids in butter fats may sometimes amount to as much as 90 per cent.³

The author has found that cocoanut oil yielded 86.43 per cent. insoluble acids,⁴ and thus infers that it could be mixed with other fats and escape detection by this method, calling attention to the fact, however, that if the soluble acids be estimated according to the method of Dupré⁵ the sophistications might at once appear.

The low figures obtained are ascribed to the volatility of lauric acid which escapes on drying the insoluble fats. By Koettstorfer's process the number of milligrams potash necessary to saponify one gram cocoanut oil was found to be 257.3 to 258.3⁶ the large quantity required being due to the presence of lauric, caproic, caprylic, and capric acids. It is, therefore, possible to mix oleomargarine and cocoanut oil in such a manner as to produce results similar to those given by pure butter. This is shown by the following figures:

Cocoanut oil.	Oleomargarine.	
<i>Per cent.</i>	<i>Per cent.</i>	<i>Milligrams.</i>
49.3+	50.7 required of KOH	220.0
70.2+	29.8 required of KOH	234.9
53.1+	46.9 required of KOH	223.6
75.9+	24.1 required of KOH	234.9

¹ *Op. cit.*, pp. 31, 62, 63.

² Analyst, 1885, p. 224 *et seq.*

³ Fleischmann and Veith, Zeit. Anal. Chem., 1878, p. 287; Kretschmar, Ber. Chem. Gesel., vol. 10, p. 2091; Kuleschoff, Wag. Jahresbericht, 1878, p. 999; Jehn, Archiv der Pharm., vol. 9, p. 335; De la Source, *Ibid.*, vol. 12, p. 929.

⁴ Chem. News, vol. 50, p. 268.

⁵ Analyst, 1877, pp. 87 and 114.

⁶ Valenta, Ding. Poly. J., vol. 249, p. 270; Moore, Chem. News, *loc. cit.*

The oleomargarine used required 193.5 milligrams KOH per gram.¹ The iodine method of Hübl² was also tried with a mixture of cocoanut oil and oleomargarine, &c., and numbers obtained which come within the limits for pure butter.

Thus oleomargarine 55 per cent. + cocoanut oil 45 per cent. required 35.5 of iodine per 100 grams, and lard 40 per cent. + cocoanut oil 60 per cent. required 32.2 of iodine. In samples of butter the iodine numbers found by Hübl varied from 26.8 to 35.1.

By Reichert's method, however,³ the presence of cocoanut oil mixed with butter and oleo is at once detected. Thus a mixture of 50 per cent. butter, 27.5 per cent. oleomargarine, and 33.5 cocoanut oil gave by Hehner's method 89.50 per cent. insoluble acids; by Koettstorfer's method, 227.5 mg. KOH; by Hübl's method, 35.4 per cent. iodine factor; by Reichert's method, 8.7cc. $\frac{N}{10}$ soda solution.

Pure butter requires by Reichert's method about 13cc., $\frac{N}{10}$ alkali to neutralize the volatile acids distilled over, while cocoanut oil in similar circumstances requires only 3.7cc. Little evidence is forthcoming in respect of the use of cocoanut oil as an adulterant of butter. It has been mentioned as an adulterant of lard⁴ and Dietsch⁵ mentions it as a compound of "Schmalzbutter." In attempts to use it as an adulterant of butter no great success was secured, since the oil not having been properly purified made the butter unpalatable. The smell and taste of the oil can be removed by a patent process of Jeserich and Meinert⁶ which consists in treating the oil with superheated steam and saponifying any free fatty acids by calcined magnesia. The author closes his paper by recommending Reichert's process as superior to all others in examining for the purity of butters.

USE OF COTTON-SEED OIL AS A BUTTER ADULTERANT.

Cotton-seed oil is used largely as an adulterant for lard and butter. It has saponification equivalent of 285 to 296 and specific gravity at 99° C. .872, pure butter fat at the same temperature being .868.

Its further properties are thus described by Allen:⁷

The oil as expressed from the seeds contains in solution, often to the extent of 1 per cent., a peculiar coloring matter, which is characteristic of this oil and its seed, and which gives the oil a ruby-red color, sometimes so intense as to cause the oil to appear nearly black. Crude cotton-seed oil gives a very bright red coloration with strong sulphuric acid (page 59). When boiled with an alkaline solution, alcoholic potash being preferable for laboratory experiments, crude cotton-seed oil is saponified, and the resultant soap rapidly oxidizes on exposure to air, with production of a fine pur-

¹ Moore, Am. Chem. J., vol. 6, p. 416.

² Ding, Poly. J., vol. 253, p. 281.

³ Zeit. Anal. Chem. 1880, p. 68.

⁴ Analyst, 1882, p. 193.

⁵ Nahrungsmittel und Getränke, p. 212.

⁶ Wag. Jahresbericht, 1882, p. 932.

⁷ Op. cit., 2d ed., p. 112.

ple or violet blue coloration.¹ This reaction is characteristic of crude cotton-seed oil. The coloring matter causes crude cotton-seed oil to produce stains, and hence is removed by a process of refining. This is usually effected by agitating the crude oil at the ordinary temperature with 10 to 15 per cent. of solution of caustic soda of 1.060 specific gravity, when the alkali combines with the coloring matter and saponifies a portion of the oil. The mixture becomes filled with black flocks which deposit on standing² and leave the oil but slightly colored. The loss in refining is usually from 4 to 7½ per cent., but occasionally amounts to 12 or 15. Hence it is desirable, before purchasing crude cotton-seed oil for refining, to ascertain, by a laboratory experiment, what the percentage of loss is likely to be. Frequently the treatment with alkali is only carried far enough to remove the major part of the coloring matter, the oil being then boiled with a solution of bleaching powder, and subsequently treated with dilute sulphuric acid.³

Refined cotton-seed oil is of a straw or golden-yellow color, or, occasionally, nearly colorless. The density ranges from .922 to .926, and the solidifying point from 1° to 10° C. By subjection to cold and pressure a certain proportion of stearine is separated, the melting point of the residual oil being correspondingly lowered. Refined cotton-seed oil is usually very free from acid, and when properly prepared is of pleasant taste and admirably adapted for edible and culinary purposes, for which it is now extensively employed, both with and without its nature being acknowledged. It is now substituted for olive oil in some of the liniments of the United States Pharmacopœia, but its principal applications are in soap making and the manufacture of facitious butter.

ESTIMATION OF SALT.

The method employed in this laboratory since 1883 has continued to give satisfaction, and can be recommended as the best in use.

From 10 to 20 grams of the well-mixed butter or butter substitute are placed in a separatory bulb provided with a closely fitting glass stopper. Add 25 to 50cc. hot distilled water, and after shaking well allow to stand for a few minutes. The water, which has dissolved most of the salt, is now drawn off through the stoppered tube of the apparatus. Fresh hot water is added and this operation repeated until the

¹ "Cotton-seed blue" is stated by Kuhlmann to have the composition of $C_{17}H_{24}O_4$. It is amorphous, readily destroyed by oxidizing agents, insoluble in water, diluted acids, and alkalis, sparingly soluble in carbon disulphide and chloroform, but more readily in alcohol and ether, and dissolves with purple color in strong sulphuric acid. The unoxidized coloring matter of cotton-seed oil has been recently examined by J. Longmore, who, in a communication to the author, states that it is a pungent golden-yellow product, insoluble in water, but soluble in alcohol and alkaline solutions, and precipitated from the latter on addition of acids. It dyes well and perfectly fast on both wool and silk.

² The deposit thus formed, consisting of coloring and albuminous matters, alkali, and partially saponified oil, is technically called "mucilage." It is decomposed with a slight excess of acid, and the resulting dark-colored grease is heated to a temperature of 120° C. (=250°F.) with concentrated sulphuric acid, which renders insoluble the coloring matters, &c., while the impure fatty acids rise to the surface. On distilling these with superheated steam, a mixture of fatty acids is obtained, which is separated into stearic and oleic acids by pressure. The "cotton-seed stearine" thus obtained is employed for making soap and composite candles, as also for adulterating tallow, &c.

³ This method of treatment is economical, but causes the oil to acquire an unpleasant taste and smell, which cannot be removed.

volume of the wash water amounts to 250 to 500cc. By this time all the salt has been dissolved and separated from the butter.

Chromate of potassium is now added to the salt solution, and the titration is accomplished by a standard silver nitrate solution.

The amount of NaCl in butter is also determined by dissolving the fat with ether or light petroleum, and after incineration of the curd, weighing the residual ash, which is taken as the amount of salt present. This method is not to be recommended since it includes the salt found in the other mineral constituents.

Sell¹ gives the following method: Ten grams of butter are weighed into a porcelain crucible and dried at 100° C. for six hours. The melted fat, &c., is now filtered, and crucible and filter are washed with ether. The filter with its contents is then incinerated. The ash is extracted with water, filtered, and the NaCl estimated volumetrically in the filtrate.

ESTIMATION OF CURD.

The methods of estimating curd depend on the principle of first drying a weighed portion of the butter, and afterwards extracting the fat with ether or petroleum. The residual mass is then weighed and the curd determined by loss on ignition. This process is carried on in this laboratory as follows:

Five to ten grams of butter are dried at 100° C. for a few hours in a porcelain dish. The dried fat, &c., are filtered through a Gooch crucible, the contents of the dish all brought into the crucible and well washed with ether or light petroleum. The filter crucible is dried for two hours and weighed. The curd is then determined by loss of weight on ignition. A number of experiments have also been made to convert the curd directly into an ammonium compound by Kjeldahl's process. This method has not met with sufficient success to merit a recommendation to general use. This method was first tried in the laboratory in 1884.

Babcock finds this method more satisfactory.² Ten grams of the fat are treated with light petroleum, and after the fat solution has been decanted the treatment is repeated. The purified curd is then treated by Kjeldahl's process.

QUALITATIVE TESTS.

The qualitative tests employed in the detection of artificial butter are the following:

(1) Microscopic examination. This method has already been sufficiently described.

(2) Solubility in a mixture of amyl-alcohol and ether.

¹ *Op cit.*, p. 527.

² Fifth Ann. Rept. Bd. Control N. Y. Exp. Sta., p. 335.

The quantity of stearin in butter fat is small compared with that in lard, tallow, &c. On this difference of constitution Professor Scheffer¹ has based a method of analysis.

A mixture is made containing 40 volumes of rectified amyl-alcohol and 60 volumes ether of .725 specific gravity at 15° C. One gram of butter fat is dissolved in 3cc. of this mixture at 26° to 28° C. On the other hand, 1 gram lard requires 16cc. of the solvent, 1 gram tallow 50cc., and 1 gram stearin 350cc.

For the experiment take a test tube of 12cc. capacity and place in it 1 gram fat, add 3cc. of the amyl-alcohol ether mixture. After tightly corking the tube put it in a water bath of 18° C. and with frequent shaking bringing the temperature to 28° C. If the butter is pure the solution becomes perfectly clear at this temperature. If not clear more of the solution can be run in out of a burette and the additional quantity required will be some indication of the quantity or quality of the adulterant which has been used.

According to Scheffer, mixtures of pure butter and lard gave the following data:

Butter.	Lard.	Quantity of mixture required.
<i>Gram.</i>	<i>Gram.</i>	<i>CC.</i>
.1	-----	3.0
.9	.1	3.9
.8	.2	4.8
.7	.3	5.7
.6	.4	6.5
.1	.9	14.4

A trial of this method has shown that it is capable of giving valuable qualitative indications in respect of the purity of the sample under examination. I believe it is the best simple test aside from the microscopic examination capable of general application which has been proposed.

The easiest method to secure a certain weight of fats is to melt them and measure out from a pipette 1 cubic centimeter of each. The fats which do not melt easily should be stirred up thoroughly with a wire, while the temperature is raised from 18° to 28° C.

(3) Odor of the burning grease.²

(4) The insolubility of the stearate of potash in alkaline solutions.³

(5) Insolubility of tallow, lard, &c., in petroleum ether of .69 specific gravity.⁴

(6) The relative solubility of butter fats and substitutes therefor in a mixture of 50 per cent. alcohol and 66 per cent. ether.⁵

¹ Pharm. Rundsch., 1886, p. 248.

² Kunstmann. Pharm. Centralh., 1875, No. 9.

³ Gatehouse, Chem. News, vol. 32, p. 297.

⁴ Zeit. Anal. Chem., 1872, p. 334.

⁵ Husson Zeit. Anal. Chem., 1880, p. 236; Filsinger, Pharm. Centralh., 1878, p. 260.

(7) Crook¹ warms half a gram of the filtered fat in a test tube to 66° C., and adds 1.5cc. phenol, shakes and warms in water bath until the liquid is clear. On standing pure butter gives a homogeneous solution. Tallow and lard appear, however, in distinct layers.

A method somewhat similar to this was proposed in 1877 by Bach.²

The apparatus required consists of a test tube and a thermometer. The reagent is a mixture of 3 volumes ether and 1 volume alcohol of 95 per cent. and 1 gram of the butter or tallow and put in the test tube with 20cc. of the above mixture, and this is placed in water at 20° C. At this temperature pure butter is completely dissolved. Butter, however, containing lard, beef, or mutton tallow remains undissolved.

(8) Horsely³ calls attention to the perfect solubility of pure butter in ether, and that it is not precipitated from this solution by methyl-alcohol, while other common fats are thus separated at 20° C.

Leuz⁴ confirms the general results of the foregoing process.

(9) Belfield⁵ allows the fats dissolved in ether to crystallize, and distinguishes between them by their crystalline form.

(10) Paillat⁶ has found that pure butter when mixed with copper oxide in ammonia gives a turquois blue color, while a butter adulterated with margarine (?) gives a greenish tint.

(11) Dubois and Padé⁷ point out that the addition of any considerable quantity of foreign fats to butter not only changes the melting point of the fatty acids obtained, but also diminishes their solubility in alcohol.

(12) Wolkenhaar⁸ distinguishes between the different fats by means of nitric acid, which gives to cotton seed oil, palm oil, lard, sesame oil, and several others a red brown color.

For a fuller discussion of most of these qualitative tests, consult either the original articles or Sell.⁹

(13) Method of Mayer.¹⁰ This test is made as follows:

About 0.6 gram of butter fat is placed in a test tube with 12cc. water made slightly alkaline by a few drops of a solution of 2 per cent. soda, or two drops of 6 per cent. ammonia-water. The tube closed by the thumb is then well shaken, afterwards carried to a temperature of 37° C. to 40° C., with frequent shaking. The emulsion thus formed is poured into a separatory funnel. The fat is now washed several times with water at 37° C. to 40° C., the wash-water being drawn off by the stop-cock so as to maintain a constant level in the funnel. The fatty matter having thus been placed in contact with about 400cc. water, the stop-cock is so

¹ Analyst, 1879, p. 111.

² Pharm. Centralb., 1877, p. 166.

³ Chem. News, vol. 30, p. 135 and 154.

⁴ Zeit. Anal. Chem., 1880, p. 370.

⁵ Rep. d. Ver. Anal. Chem., vol. 3, p. 383.

⁶ L'Année Scientifique par Louis Figuier, 29th year, 1885.

⁷ Bul. Soc. Chim., vol. 44, p. 602.

⁸ Rep. d. Ver. Anal. Chem., vol. 3, p. 103.

⁹ Op. cit., pp. 505-509.

¹⁰ Jour. de Pharm. et de Chim., vol. 15, p. 97.

adjusted as to allow the removal of the wash-water as completely as possible. After cooling, the fatty matter remaining on the sides of the funnel is examined. If the butter be pure, there will be seen only a finely-divided mass, but the addition of a small portion of other fats will be revealed by greasy drops, which can be seen even during the progress of the washing. Natural butters made in summer require a lower temperature for the washing, viz, 35° C. to 37° C.

In most cases the microscopic test with polarized light and selenite plate combined with the solubility of the fat in the ether amyl-alcohol solutions will be found sufficient for the qualitative examination of a suspected butter.

RESULTS OF ANALYSES OF GENUINE AND SUSPECTED BUTTERS AND BUTTER ADULTERANTS.

TABLE NO. 6.—*Analyses of butter.*

Serial number.	Specific gravity at 40° C.	Water.	Insoluble acid.	Soluble acid, by washing out.	Soluble acid, by distillation.	Salt, NaCl.	Albuminoids.	Curd.	Koettstorfer's equivalent.	N Vol. 10 soda for 2.5 grams.
		<i>Pr. ct.</i>	<i>Pr. ct.</i>	<i>Pr. ct.</i>	<i>Pr. ct.</i>	<i>Pr. ct.</i>	<i>Pr. ct.</i>	<i>Pr. ct.</i>		
1742.....	.91046	13.33	88.64	4.01	4.50	2.84	.7875	1.46	254.20	12.50
1743.....	.91119	8.53	87.85	4.14	4.57	3.69	.8312	1.31	250.60	13.10
1744.....	.91032	8.57	88.65	3.52	4.78	2.81	.8750	1.30	268.50	13.50
1745.....	.91067	8.14	88.08	3.68	5.48	2.04	.5688	1.25	264.90	15.30
1746.....	.910-9	16.82	88.91	3.00	4.56	3.79	.7438	1.56	252.70	12.90
1747.....	.91244	4.59	86.60	5.02	5.51	3.41	.5250	.83	244.30	15.60
1749.....	.91165	11.44	87.50	5.49	4.61	1.48	.8312	1.14	250.10	13.10
1752.....	.91004	17.38	88.07	3.70	4.54	0.00	.4375	0.63	238.60	12.80
1759.....	.91013	13.95	87.47	4.73	4.80	0.00	.4375	0.81	249.70	13.60
1760.....	.91063	22.12	87.84	4.98	4.70	0.00	.1750	0.49	248.70	13.40
1761.....	.91067	23.46	87.47	5.27	4.99	0.00	.1750	0.59	243.00	14.10
1762.....	.91089	21.02	87.38	5.15	4.93	0.00	.2188	1.01	248.80	14.10
1763.....	.91073	11.89	87.71	4.69	4.98	2.61	.2625	1.30	244.90	14.10
1764.....	.91155	21.96	86.65	5.34	4.74	0.00	.4375	1.21	244.00	13.20
1765.....	.90958	31.55	88.09	4.45	5.02	0.57	.6125	1.83	252.00	14.30
1766.....	.91042	11.17	5.31	4.52	2.56	.4375	1.11	247.00	12.80
1768.....	.90995	7.68	87.24	5.08	5.21	5.62	.2625	0.71	247.00	14.80
1769.....	.91183	9.68	87.30	5.94	5.05	4.09	.5230	1.37	244.10	14.30
1770.....	.91069	7.35	88.14	5.05	4.47	5.28	.4375	0.91	252.10	12.70
1771.....	.91079	12.28	87.00	5.37	4.93	3.69	.4813	1.08	246.40	14.00
1772.....	.91093	8.89	87.21	5.47	5.26	3.18	.3063	1.03	245.10	14.90
1773.....	.91064	18.75	86.68	4.75	4.03	0.00	.7000	1.41	260.70	11.40
1775.....	.91034	9.87	87.58	5.17	4.56	4.83	.4375	1.12	251.80	12.90
1776.....	.91239	10.84	86.61	5.42	4.45	3.12	.4375	0.97	250.90	12.70
1777.....	.91031	12.28	88.48	4.66	3.92	5.79	.7438	1.43	236.50	11.10
1781.....	.91010	7.26	3.97	6.42	.4375	1.43	247.10	13.20
1782.....	.91112	12.32	87.23	4.24	6.53	.5250	2.02	245.40	13.60
1783.....	.91082	6.93	87.59	3.92	3.92	.5250	1.33	248.40	12.50
1785.....	.91186	8.29	87.10	4.48	5.11	.4375	1.16	247.50	14.50
1789.....	.91061	8.44	87.73	3.91	3.15	.7000	1.42	246.60	12.60
1790.....	.91080	4.44	87.85	4.41	1.81	.7000	1.02	251.50	13.90
1792.....	.91106	13.67	88.25	3.47	7.10	.4375	3.10	240.20	12.30
1795.....	.91136	8.22	87.75	4.18	4.37	.5125	1.34	240.70	14.50

TABLE NO. 7.—*Analyses of doubtful butters.*¹

1748.....	.90968	7.45	89.45	3.61	4.60	2.64	.7443	1.41	252.80	13.10
1757.....	.90964	11.30	89.44	3.54	4.25	5.28	.5688	1.63	253.60	12.10
1758.....	.90987	12.12	87.60	4.71	4.54	0.00	.4375	0.93	251.50	12.90
1767.....	.90974	10.90	88.68	4.73	4.45	2.16	.4813	1.33	249.70	12.60
1774.....	.90972	29.84	87.82	4.84	4.27	0.00	.9625	1.86	260.10	12.10
1779.....	.90947	11.59	88.01	3.16	5.00	.8750	1.56	250.60	12.50
1780.....	.90964	10.66	88.42	3.02	5.40	.8750	1.58	250.70	11.60
1793.....	.90938	8.50	88.00	3.34	13.00	1.12	253.50	12.30
1794.....	.90965	9.06	88.50	3.44	2.84	.4375	0.98	252.00	11.70

¹ These samples were bought for pure butter, but, on analysis, proved to contain adulterants.

TABLE NO. 8.—*Analyses of butter substitutes.*

Serial number.		Specific gravity at 40° C.	Water.	Insoluble acid.	Soluble acid, by washing out.	Soluble acid, by distillation.	Salt, NaCl.	Albuminoids.	Curd.	Koettstorfer's equivalent.	N Vol. 10 soda for 2.5 grams.
			<i>Pr. ct.</i>	<i>Pr. ct.</i>	<i>Pr. ct.</i>	<i>Pr. ct.</i>	<i>Pr. ct.</i>	<i>Pr. ct.</i>	<i>Pr. ct.</i>		
1750	Lard90538	0.00	92.59	0.41	0.08	0.00	.0875	Trace	294.30	0.20
1751	Beef suet90158	0.00	92.59	0.22	0.04	0.00	0.01	296.90	0.10
1753	Oleomargarine90490	9.34	93.59	0.12	0.25	3.64	.3500	0.63	274.00	0.70
1754	Neutral lard90369	7.42	90.00	0.20	0.10	0.40	0.02	270.50	0.30
1755	Creamery butter- ine ¹90569	11.69	92.90	1.16	1.53	2.39	.3063	0.74	274.80	4.30
1756	Oleo fat ²90237	14.23	93.35	0.10	0.08	0.97	0.60	286.10	0.20
1787	Country print....	.90561	14.45	93.72	0.09	2.42	.8750	1.82	281.10	1.90

¹ 40 butter fat, 15 oleo fat, 30 neutral lard.² Average 40 pounds per fat steer.

ANALYTICAL RESULTS.

The butters in table No. 6 were bought in open market and accepted as genuine on the results of the analysis. Some of these, however, ought justly to be classed in Table No. 7, as of doubtful purity. In quite a number of cases the number of cubic centimeters of decinormal alkali required to neutralize the distillate from 2.5 grams of the fat was less than 13. Nos. 1742, 1746, 1752, 1766, 1770, 1773, 1775, 1776, 1777, 1783, 1789, and 1792 come under this category. In all these cases, however, except 1755 and 1768, the specific gravity is above .910 at 40° C., and it would not be safe to condemn a butter as adulterated which had that specific gravity, unless the microscope should reveal crystals of foreign fat. In these samples such was not the case.

In the two cases mentioned, where the specific gravity fell below .910, there are other reasons for thinking the samples pure. In 1765 the percentage of soluble acid, by Reichert's method, is high, viz, 5.02. In 1768 it is still higher, viz, 5.21. With such proportions of soluble acid it would not be possible to condemn the samples as adulterated on the evidence of the specific gravity alone.

On the other hand, when the percentage of soluble acid is low, as in 1777, the specific gravity and saponification equivalent prevent the classification of the sample among the doubtful butters. Nevertheless, should such a sample show with polarized light and a selenite plate bi-refractive crystals, it would be a strong presumptive evidence of adulteration. In any case, such a sample as 1777 would present numerous difficulties to the analyst, especially if he were called to testify in respect to its purity.

In Table No. 7 similar difficulties are encountered. The specific gravities are uniformly low. On the other hand, the percentage of insoluble acids are only suspiciously high in two instances, viz, 1748 and 1757. In the first of these instances, however, the soluble acid is above the limit of suspicion. The saturation equivalent is uniformly rather high,

but not above the range of pure butters. While the butters are classed for convenience as "doubtful," they could not be so proved before a court on the chemical evidence alone.

In Table No. 8 we have plain sailing. All analytical data show the fats of the samples examined are not butter. Since the adulteration of butters with less than 30 per cent. of a cheaper fat could scarcely prove profitable, the chemist should be careful not to condemn a suspicious sample, if its purity be attested by any one of the processes employed in the examination, unless some one test shows it to be undoubtedly adulterated.

In the foregoing study of methods of analysis I have not attempted to give a complete citation of all the papers which have been written on this subject. A very complete bibliography of the subject up to 1882 is given by Caldwell,¹ and in the work of Sell.²

The probability of the detection of an adulterated butter by the physical and chemical processes described in the foregoing pages is very great.

In the order of value the quantitative processes employed may be arranged as follows: (1) Determination of volatile acids by distillation. (2) Determination of specific gravity. (3) Determination of the saponification equivalent. (4) Determination of the insoluble acids. (5) Determination of the melting point.

¹ Second Ann. Rept. N. Y. S. Bd. of Health, pp. 544-7.

² Arbeit a. d. Kaiserlichen Gesundheitsamte.

EXAMINATION OF MILK.

The adulteration of milk in this country consists usually either in the removal of cream or the addition of water.

Without making any attempt whatever to notice the prolific literature of this subject, which has accumulated during the past few years, such portions thereof as seem to be most helpful in the work of analysis will be cited. Those who care to study the subject in greater detail are referred to the periodical literature, especially to the "Analyst" and "Milch Zeitung."

The constituents of milk which are to be determined by analysis are (1) water; (2) sugar; (3) nitrogenous constituents; (4) ash, and (5) fat.

Water.—The simplest method for estimating water in milk consists in evaporating one or two grams in a flat platinum dish. The larger the diameter of the dish the quicker and more accurate will be the results.

If larger quantities of milk be used or the dish have not a flat bottom, the film which forms over the surface of the milk during evaporation will prevent complete desiccation. To avoid this many plans have been proposed. The milk may be mixed with gypsum, and then a larger surface be exposed and more rapid and complete drying secured.

Instead of gypsum, sulphate of barium, pure quartz sand, sulphate of strontium, and powdered glass have been used. All of these methods are capable of giving fairly accurate results when properly conducted.

The addition of acetic acid or alcohol to coagulate the albuminous matter before desiccation has been largely practiced, but Gerber and Radenhausen have shown¹ this treatment is without influence on the results. Jenks has also shown² that simple evaporation without any treatment whatever gives results which agree well with those obtained by using sand.

In fifty determinations the maximum and minimum difference between the two methods was only .14 per cent. and the mean difference .003 per cent.

Babcock³ has proposed an ingenious and accurate method of determining the water in milk:

About two grams of rather coarse asbestos are placed in a platinum evaporator of 30cc. capacity, ignited and weighed. Five cubic centimeters of milk from the pipette,

¹ Bied. Centralblatt, 1876, p. 22.

² Chem. Centralblatt, 1882, p. 13.

³ Second Ann. Rept. Bd. Control N. Y. Exp. Sta., pp. 167-8.

previously weighed, is run into the evaporator and the pipette weighed again. The milk in the evaporator is then dried at 100° C., until the weights taken one-half hour apart do not vary more than a milligram from each other.

The asbestos serves as an absorbent of the milk and presents a large surface which greatly facilitates the drying. For this purpose asbestos is much to be preferred to sand or any fine powder which requires frequent stirring for complete desiccation. When a number of analyses are to be made in succession, a second portion of milk may be dried in the same asbestos with advantage. In the series of analyses made during the feeding experiments the morning's and evening's milk were dried together in this way. The dried residue may be ignited for ash.

The figures given for solids in all analyses made during the year have been determined in the above manner. The solids may, however, be found with equal accuracy and in much less time by the method given below.

In the bottom of a perforated test-tube, such as is used in the estimation of the fat in fodders, is placed a tuft of clean cotton. The tube is then filled three-quarters full of ignited asbestos and a plug of cotton inserted to prevent the escape of loose fibers of asbestos. The asbestos must be slightly pressed together so as to leave no large spaces. The tube and contents are weighed, the plug of cotton carefully removed, and five grams of milk, from the weighed pipette, described before, run into it and the plug of cotton replaced. The tube, connected at its lower end by a rubber tube and adapter with a filter pump, is placed in a drying oven at 100° C. and a slow current of dry air drawn through it till the water is completely expelled, which in no case requires more than two hours.

Since the publication of the method of Adams for the estimation of fat, which will be given further on, I have made some attempts to estimate the water by drying the milk on long strips of asbestos paper, which are rolled up while still hot and weighed after cooling in a dessicator. I have not yet secured an asbestos paper sufficiently bibulous to make this method completely successful. But it has the advantage of being very speedy, since on so large a surface exposed for two or three minutes to a temperature of 100° to 105° C. over a sand bath the water is completely evaporated.

An indirect method of estimating the water from the specific gravity has been prepared by Behrend and Morgen¹ by the formula—

$$S^2 = \frac{S(V-A)}{V - S^1}$$

in which S = specific gravity of the milk, S² = specific gravity of the milk free of fat, S¹ = specific gravity of the milk fat = .94, and V = volume taken = 100cc.

Numerous tables are given by the authors to show the agreement between the calculated percentage of fat and total solids obtained by the above formula and the gravimetric determinations.

Another indirect method of determining the quantity of water in milk consists in measuring the quantity of finely-pulverized common salt a given volume of it will dissolve.

This procedure was proposed by Reichelt.²

The apparatus consists of a glass vessel 24cm. high. The upper part has a diameter of 2.5cm. and the lower of 8mm. On the under side is a

¹ Jour. Landw., 1879, p. 249.

² Bayerish Kunst und Gewerbeblatt, 1860, p. 706.

scale marked to 45° C. The principle of the apparatus is based on the fact that at 30° to 35° C., 100 parts of water will dissolve 36 parts of salt. The operation is carried on as follows: Mix 62.5 grams of milk with 20.25 grams of salt and add 15 grams of litmus tincture, saturated with salt, to color the milk. Raise the temperature to 30° to 35° C., shake thoroughly, and then place the apparatus so that all the undissolved salt will fall into the under-graduated stem of the apparatus. Each degree of the scale corresponds to 62.5mgr. of the salt. The part undissolved subtracted from the total quantity will give the quantity dissolved, from which the quantity of water is easily calculated.

The lactometer of Geissler¹ is too complicated for ordinary use, and the method of estimating the water content of milk by measuring the volume of whey filtered from the coagulated albumens proposed by Zenneck² does not afford sufficiently exact results to merit further description.

SPECIFIC GRAVITY.

The specific gravity of a milk diminishes as its content of fat increases, and hence within certain limits it may be a valuable index of the character of the sample under examination.

When the cream has been removed, however, the specific gravity may be reduced to that of normal milk by the addition of water, and then the determination of the specific gravity alone is not a certain method of detecting adulteration, yet it is a valuable indication and should always be determined.

This determination may be made by any of the methods already denoted for fats and oils or by a hydrometer. Since the use of this latter instrument (lactometer, lactodensimeter) is easy and speedy, it is generally employed instead of the slower but more exact procedure with a pycnometer.

Martin³ found the average specific gravity of the milk from fifty cows from E. B. Brady's farm, Westchester, N. Y., to be 1.03101. From another lot of thirty-one cows, farm of Peter Knox, it was 1.03149; from sixteen cows, farm of George Nelson, 1.03175.

Jenkins⁴ makes the following observations respecting the values of the specific gravity determination:

A consideration of the observations noticed above brings us to the following conclusions with regard to the value of *total solids*, and of *specific gravity*, as criteria for judging of the quality of milk.

We have seen that pure herd-milk shows very wide variations in its content of solids and fat, and variations less striking in its specific gravity. No instance appears to be on record where a competent observer has found for the mixed milk of a number of healthy cows a specific gravity less than 1.029, and we may conclude with certainty that milk which falls below that density has been watered.

¹ Ber. Chem. Gesel., Vol. 10, p. 1272.

² Vieth, Milchprüfungsmethoden, p. 87.

³ Fourth Ann. Rept., N. Y. State Bd. of Health, pp. 429 *et seq.*

⁴ Rept. Conn. Exp. Sta., Vol. 10, pp. 88, 89.

As evidence of watering simply, specific gravity furnishes by far the most satisfactory test, and if 1.029 is adopted as a minimum, no pure milk will be condemned. In some cases moderately watered milk may escape detection.

If we will establish a minimum limit for the percentage of solids and fat which shall in no case condemn pure milk in any locality, we shall have to make it absurdly low, and thus offer a premium on watering milk of good quality.

QUANTITY OF WATER OR DRY SOLIDS IN MILK.

The law of Massachusetts fixes the legal maximum of water in milk at 87 per cent. The quantity, however, varies within large limits, and it is manifestly unjust to condemn a milk as adulterated when it has more than 87 per cent. water.

The chief factors which cause a healthy milk to vary in its percentage of solids are length of time the cow has been in milk, the season of the year, and consequently the character of the food of the animal. On this point the Report of the Massachusetts State Board of Health¹ makes the following observations:

The statutes prescribe a fixed and definite standard for commercial milk. Milk not containing 13 per cent. of solids is deemed to be adulterated under the law. It is often urged that, under such a standard, milk as obtained direct from the animal does not always conform to the requirements of the law. While this is true, it is also evident that a standard established at the minimum of quality, or that of the poorest milk obtained under the worst conditions, would admit of the sale of a very large quantity of adulterated milk.

It is possible to produce from inferior animals, under unfavorable conditions, such as impoverished diet, bad care, extreme age or youth, milk somewhat below the legal requirement. This ought not to be an argument for the reduction of the standard to include occasional cases of the lowest quality.

Mixed milk contains a greater amount of solids than its minimum constituents. Hence, the milk producer or dealer will find it a safe rule to sell mixed milk only, especially when his herd contains one or more animals producing milk of a poor quality. In the 40-quart cans of the Housatonic Valley, filled for the New York market, the milk must necessarily be a mixture from several animals, but in the case of the usual 2-gallon can, so largely in use throughout the larger part of this State, the contents may be often that of two or three animals only, and it occasionally may represent a single animal.

Under the Massachusetts law a rigid inspection of the milk sold in all the large cities is made, and the character of the milk is described in the following summary of the report of Dr. Harrington, milk inspector for Boston.²

During the year just ended I have received from the inspectors of the board, and from other sources, 1,759 samples of milk, which number includes samples from all of the cities and many of the towns of Eastern Massachusetts. They have been arranged in classes, according to their respective sources, to wit: (a) Samples from shops; (b) samples from wagons; (c) samples from producers (direct); (d) samples from unknown sources; (e) samples of known purity.

Among such a number, taken in most cases at random, there must necessarily be very many which, on inspection alone, are evidently pure, and which, on analysis, would yield figures above the standard fixed by law. The employment of the lactodensimeter, together with the Feser lactoscope, will, after a little practice, enable

¹ 1884, p. 116.

² Rept. Mass. S. Bd. of Health, 1884, pp. 145, 14

one to separate the good from the indifferent and poor samples, and in this way the good samples may, unless there be some reason for a full analysis—such, for instance, as unusual richness, averages, &c.—be passed on inspection. More than half of the samples submitted to me were good samples, and of the whole number 569 were passed as above, leaving 1,190 which were subjected to analysis. Of these latter there were 391 which were above, and 799 which were below, the statute standard.

Considering as above the standard the 569 samples which were passed on inspection, we have 960 above and 799 below, which is a very decided improvement over the milk supply of a year ago. A large proportion of those below the standard were not what would be considered as of very inferior quality; nearly one-half of those below the standard of 13 per cent. of solids were above 12 per cent., which fact of itself is evidence of a great improvement in the general supply.

Dr. E. W. Martin¹ has made a thorough study of milk adulteration, of which I give the following abstract:

Having made from time to time analyses of milk from cows of all breeds and kept under all conditions, of various ages, and at different times of the year, I found the percentage of the maximum, minimum, and average constituents to be:

Constituent.	Maximum.	Minimum.	Average.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Water.....	82.04	87.87	87.5
Fat.....	7.59	2.78	3.3
Sugar.....	5.39	4.60	4.4
Caseine.....	4.34	4.30	4.1
Salts.....	.71	.65	.7
Total solids.....	17.96	12.13	12.5
Solids not fat.....	10.37	9.35	9.2

Of the minimum amount of fat given above, only three cows were found giving milk so poor in fat, and their food and surroundings were of the poorest kind.

During the year 1883 I made many analyses of milk taken from the cans while being shipped to market, and the average percentage of the constituents of the samples taken were:

	<i>Per cent.</i>
Water.....	87.5
Fat.....	3.2
Sugar.....	4.4
Caseine.....	4.1
Salts.....	.7
Total solids.....	12.5
Solids not fat.....	9.3

From the foregoing results it is fair to assume that in average milk we should have at least 3 per cent. of fat, 9.2 per cent. of solids not fat, and 12.2 per cent. of total solids.

The State Board of Health of New Jersey have fixed the minimum amount of total solids at 12 per cent. and the maximum amount of water at 83 per cent.

In Massachusetts the law fixes a chemical standard of purity; it reads: "In all cases of prosecution, if the milk shall be shown upon analysis to contain more than 87 per cent. of water or to contain less than 13 per cent. of milk solids it shall be deemed for the purpose of this act to be adulterated."

This standard has been fixed from analyses by Sharples, Babcock, and others, as follows:

Analysts.	No. of cows.	Total solids.
Sharples.....	22	14.49
Babcock.....	8	14.55
Vaughn.....	58	14.08
Newton.....	24	14.26

¹Fourth Ann. Rept. N. Y. S. Bd. of Health, pp. 429 *et seq.*

ESTIMATION OF FAT.

Gravimetric.—The percentage of fat in milk is the best criterion of its purity, although it is not impossible to make an emulsion with an added fat or oil after the natural fat of a milk has been removed.

For determining the quantity of fat in a milk by weight it is necessary to evaporate the sample to dryness and dissolve the fat by ether or a light petroleum.

Since, if the milk be evaporated in bulk, portions of the fat will be occluded by the other solids, it is necessary, in order to secure a total solution of the fat, to distribute it over some inert substances. Those already mentioned used for determining water, may also be employed for the fat analyses.

We use thin glass dishes (*schälchen*), in which the milk is dried in a thin film, or on sand, gypsum, or asbestos. The dish and its contents are then rubbed up in a mortar and transferred to a continuous extraction apparatus. The one employed is a modification of the continuous extractor made by Mr. A. E. Knorr, in which the return siphon is placed wholly within the extraction tubes, thus making the apparatus more compact and less liable to get broken.

The different methods of manipulation heretofore employed for the gravimetric determination are so well known that I will not describe them further, but pass at once to the consideration of a new process, which I have already tried sufficiently to show its merits over all others.

ADAMS'S METHOD.

Adams's method of estimating fat in milk¹ differs from the ordinary gravimetric methods solely in the preparation of the fat for extraction. Instead of drying the fat in the usual way the milk is absorbed by bibulous paper. It is unnecessary to state that this paper must first be thoroughly exhausted by the solvent which is used to dissolve the fat.

The kind of paper and the method of using it first proposed by Adams, are as follows:

As for material, the only extra article is some stout white blotting-paper, known in the trade as "white demy blotting mill 428," weighing 38 pounds per ream. This should be in unfolded sheets, machine-cut into strips $2\frac{1}{2}$ inches wide and 22 inches long; each sheet in this manner cuts into seven strips.

I have tried other papers, but none have answered so well as this; it is very porous and just thick enough. Each of these strips is carefully rolled into a helical coil, for which purpose I use a little machine, made by myself, consisting of a stout double wire, cranked twice at right angles, and mounted in a simple frame. One end of the strip being thrust between the two wires, the handle is turned, and the coil made with great facility. This may be done, for the nonce, on a glass rod, the size of a cedar pencil. Two points have to be carefully attended to: the paper must not be broken, and the coil must be somewhat loose, the finished diameter being a little under an inch. I am in the habit of rolling up a considerable number at a time and

¹ *Analyst.*, 1885, pp. 46 *et seq.*

placing each within a brass ring as it is rolled, inscribing on one corner with a lead pencil its own proper number.

These coils are next thoroughly dried, and I need hardly say the accuracy of the process depends upon this drying. This can be satisfactorily done in an ordinary air bath at 100° C., providing the bath be heated properly and the paper kept in it long enough. I found the common way of heating the thin bottom of the bath with a single jet not to answer. My bath is placed upon a stout iron surface, which is heated by a large ring of jets; in this way the heat is evenly distributed over the whole of the bottom of the bath, and the papers, which are put in a cage frame of tinned iron wire 5 by 2½ inches and divided into eight partitions, get evenly and completely dried, if allowed to remain in the bath all night, and weighed in a weighing tube next morning, and their weights having been registered according to their numbers, stored away ready for use, as follows:

The milk to be examined is shaken, and with a pipette 5cc. are discharged into a small beaker 2 inches high by 1½ diameter, of a capacity of about 30cc. weighing about 12 grams. This charged beaker is first weighed, and then a paper coil gently thrust into the milk very nearly to the bottom. In a few minutes the paper sucks up nearly the whole of the milk. The paper is then carefully withdrawn by the dry extremity of the coil and gently reversed, and stood, dry end downwards, on a clean sheet of glass. With a little dexterity all but the last fraction of a drop can be removed from the beaker and got on the paper. The beaker is again weighed, and the milk taken got by difference. It is of importance to take up the whole of the milk from the beaker, as I am disposed to consider the paper has a selective action, removing the watery constituents of the milk by preference over the fat.

The charged paper is next placed in the water oven on the glass plate milk-end upwards, and rough-dried. Mismanagement may possibly cause a drop to pass down through the coil onto the glass. This accident ought never to occur; but if it does, it is revealed in a moment by inspection of the surface of the glass, and the experiment is thereby lost.

In about an hour it is rough-dried and in a suitable condition for the extraction of the fat.

The method of Adams has been thoroughly tried by the English chemists, and has received the approval of the English Society of Public Analysts. It gives uniformly about .2 per cent. more fat in normal milk than the ordinary gravimetric methods.

In this laboratory we use the following modification of the process:

The blotting paper is replaced by thick filtering paper cut into strips 2 feet long and 2.5 in. wide. These are thoroughly extracted by ether or petroleum.

One end of the strip of paper being held horizontally by a clamp or by an assistant, 5cc. milk is run out by a pipette from a weighing bottle along the middle of the strip of filtering paper, being careful not to let the milk get too near the ends of the paper, and to secure an even distribution of it over the whole length of the slip. The pipette is replaced in the weighing bottle and the whole reweighed, and thus the quantity of milk taken is accurately determined. The strip of paper is now hung up over a sand bath in an inclosed space high enough to receive it where the air has a temperature of 100° C. (circa). In two or three minutes the paper is thoroughly dry. It is at once, while still hot, rolled into a coil and placed before cooling in the extraction apparatus already described.

The fat is dissolved by ether or petroleum, collected in a weighed flask, and, after thorough drying, weighed. I have already mentioned that by the use of asbestos paper I had hoped to be able to estimate the moisture in milk, but so far have not achieved the success which I believe is possible.

Of all the methods for the gravimetric determinations of fat in milk, I do not hesitate to say that the Adams method, properly carried out, is the best.

SOXHLET'S AREOMETRIC METHOD OF ESTIMATING FAT IN MILK.¹

Caldwell and Parr² call attention to the difficulty which is often experienced in using Soxhlet's areometric method for the estimation of fat in milk. They say, speaking of the determination of fat by the lactobutyrometer: "But in this case, while the butyrometer gave tolerable results, Soxhlet's method failed entirely; even after standing five hours the layer of ether fat solution in the mixing bottle, which should be over a centimeter thick, was hardly a millimeter thick."

This experience is so much in harmony with my own that I thought it would be of interest to call attention to some of the difficulties encountered in working with Soxhlet's method.

Soxhlet's original paper was published in "*Zeitschrift des Landwirtschaftlichen Vereins in Bayern*," in 1880.

It rests upon the assumption that an alkaline milk shaken with ether will give all its fat to the ether, and this solution, being lighter than the rest of the mixture, will collect at the top, where it can be separated and its specific gravity determined.

The reagents used are ether saturated with water and a solution of caustic potash containing 400 grams to the litre.

The milk and reagents having been brought to a temperature of 17.5° C. are measured into a flask (I use an ordinary pint beer bottle), with pipettes furnished with the apparatus; 200cc. milk, 10cc. of potash solution, and 60cc. of the aqueous ether are the quantities to be employed. The milk is first placed in the flask and to this the potash solution added and shaken vigorously. Afterwards the ether is added and the shaking continued for one minute longer. The bottle is then put into water at a temperature of 17.5° C. and gently struck on the table in a vertical position at intervals of half a minute for fifteen or twenty minutes. At the end of this time the ether-fat solution has collected at the top, whence it is passed to the areometric cylinder by means of the rubber bulb blowing apparatus shown in figure 1.

Water is now added at a temperature of 16° C. to 18° C. to the outer cylinder, and after the temperature has become constant the density of the ethereal solution is read on the scale of the areometer. At the same time the temperature is read from the delicate thermometer attached

¹ H. W. Wiley, *Journal of Analytical Chemistry*, vol. 1, no. 2.

² *Am. Chem. Jour.*, vol. 7, p. 245.

to the areometer. The areometric degree is to be increased or diminished by the difference between the observed temperature and 17.5°C . as the former is above or below the latter. The percentage of fat is taken from a table which gives the numbers representing it for all degrees of the Soxhlet's scale between 43 and 66 for whole milk, or 2.07 to 5.12 per cent.; and for skimmed milk from 21.1 to 43, or from 0.00 to 2.07 per cent. It is thus seen that the scale includes all percentages of fat from nothing to 5.12. If a milk contain more than the latter percentage of fat it must be treated with a certain proportion of water before it can be examined by the Soxhlet's method.

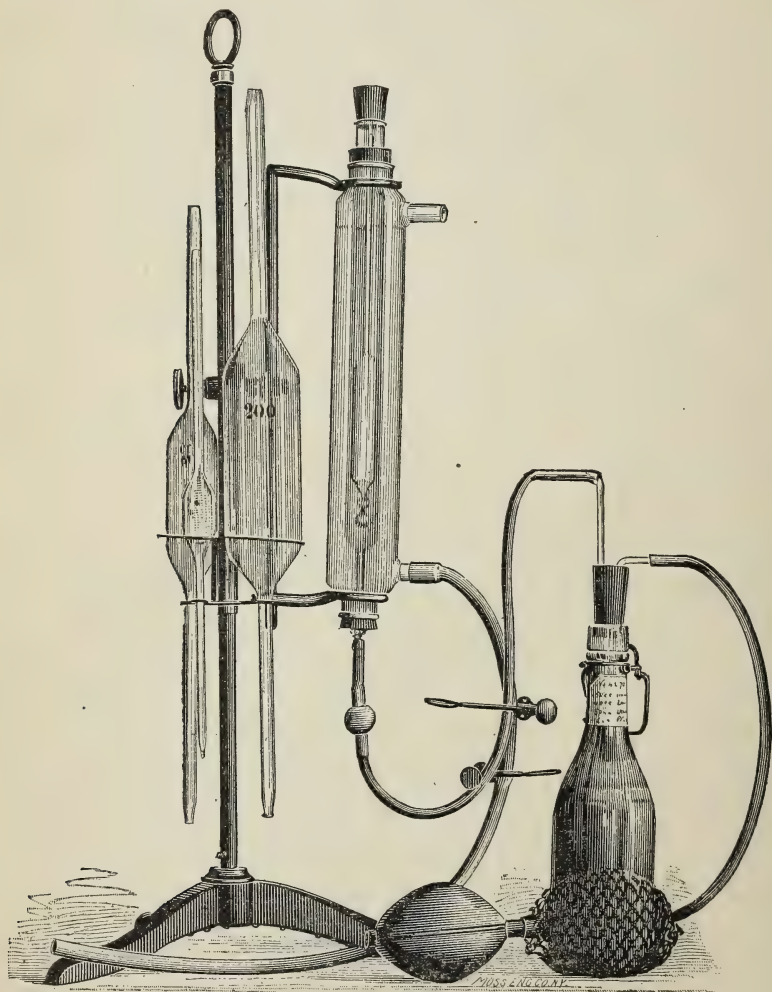


FIG. 1.

Both Soxhlet in his original paper and Liebermann¹ affirm that the ether retained in the form of an emulsion in the lower part of the liquid

¹ Zeit, Anal. Chem., 1884, p. 478,

in the flask does not contain a trace of fat. It is, therefore, necessary to assume that the emulsion has always the same proportion of ether, otherwise there would be variations in the density of the clear supernatant solution. This may be entirely true with those milks which permit the ether solution to separate readily, but where the separation is difficult and a great deal of time is required for it to take place, it is possible that this assumption may not hold good.

Liebermann, who having once condemned Soxhlet's method, on account of the slow separation of the ether solution, introduced a modification of gently shaking the mixture and then pronounced it successful. He says¹: "Therefore the manner of making the first shaking with ether is not immaterial. The shaking must not be violent (Soxhlet says 'schütteln eine halbe Minute kräftig'), but, nevertheless, always sufficiently so. The light vertical blows must also be skillfully applied. In fact, these are things which can only be attained by the skilled touch coming from experience."

It appears from the above, and my own experience fully convinces me of the truth of it, that in order to secure a proper separation of the ether fat solution, in all cases, one must be possessed of the art of leg-erdemain.

After giving the preference to his own volumetric method Liebermann ends his paper by the remark: "But if, on the contrary, the method of Soxhlet is employed, it will usually happen that a given milk must be examined twice, once to see whether the original method is applicable, and again, in case of failure, with the necessary modifications."

I think it will be readily agreed that such a method can find no place in the examination of the milk of individual cows for each sample of which a special modification of the method would have to be made.

Schmoeger² calls attention to the shortcomings of Soxhlet's method when milk poor in fat or skimmed milk is used. He says milk containing under 2 per cent. of fat must be evaporated with gypsum before it will give up its fat to ether. Yet it must not be forgotten that gypsum will give up something to ether, also. Schmoeger obtained from 20 grams of pure ignited gypsum 5mg. and from 20 grams sea sand 2mg. of extract. These facts must be taken into consideration when the gravimetric comparisons of fat per cents with the areometric results are made. Certain variations in the method of making these gravimetric determinations have lately come into use, which promise some improvement. Babcock³ substitutes ignited asbestos for sand or gypsum. This, in my opinion, is a great improvement. Babcock⁴ also dispenses with the evaporating dish and uses a tube open at both ends filled with as-

¹ Zeit. Anal. Chem., *loc. cit.*

² Bericht ueber die Thatigkeit des Milchwirthschaftlichen Instituts zu Proskau, as quoted in Zeit. Anal. Chem., 1885, p. 130.

³ Second Ann. Rept. Bd. Control N. Y. Exp. Sta., p. 167.

⁴ *Loc. cit.*

bestos, with a plug of cotton at each end. The milk is absorbed by the asbestos, the tube is then placed in a steam bath and a slow current of air drawn through it by an aspirator. In two hours the desiccation is complete. The method gives satisfactory results. It is inconvenient, however, to work a large number of samples at once by this method. After drying, the tube is placed in a continuous extractor and the fat removed by ether. I have used the following modification of the gravimetric method. The evaporation is made in a schälchen half filled with fine, pure asbestos. About 5 grams of milk are taken for each determination. The water is driven off at 100° C. and after the total solids have been noted by weighing, the asbestos is removed to the extraction tube. The schälchen is then finely ground in a mortar, transferred to the extraction tube and the mortar and pestle thoroughly washed with ether. The extraction is then made in the usual way.

Adams¹ proposes the use of blotting paper rolled into a helical coil as the absorbent of the milk for the purpose of drying and extraction of the fat. Johnstone² modifies this method by using disks of blotting paper held in a circular platinum dish. Dr. Vieth³ hesitates to approve the blotting paper method on account of the fact that blotting paper itself gives up a considerable extract to ether.

Allen and Chattway⁴ recommend certain modifications in Adams' method, consisting in a peculiar method of winding the coil of blotting paper.

Thompson⁵ proposes the use of filtering paper instead of blotting paper. I have had this method tried in the laboratory by Mr. Knorr and with pleasing results. Care must be taken, however, to use only filtering paper which has been previously thoroughly extracted. It was found that the filtering paper necessary to absorb 5 grams of milk was afforded by a piece 24 inches in length by 2.5 inches in breadth. This paper extracted with ether gave the following residues:

	Milli-grams.
No. 1.....	11.4
No. 2.....	13.4
No. 3.....	12.0
No. 4.....	10.5
No. 6.....	12.2
No. 7.....	20.9
No. 8.....	15.5

Since the tables of the Soxhlet method are based on the old method of extraction they will have to be revised for the new data given by the Adams method.

One great advantage of the Adams method as modified by Thompson I have found to consist in the rapidity with which the preliminary dry-

¹ Analyst, 1885, p. 48.

³ *Ibid.*, 1885, p. 86.

⁵ *Ibid.*, 1886, p. 73.

² *Ibid.*, 1885, p. 83.

⁴ *Ibid.*, 1886, p. 71.

ing is accomplished. I have been able to put 5cc. of milk on a strip of paper, hang it over a sand bath and have it rolled and in the extractor within five minutes.

I mention this to show that even in the matter of gravimetric determinations by which the areometric method is finally judged, there is still a certain limit of variability.

I will return now to the subject more immediately under discussion. Schmoeger further says¹ that with skimmed milk, buttermilk, and such milks as have stood twenty-four hours on ice the ether-fat solution separates difficultly or not at all. To avoid this he recommends, after the addition of the potash, fully five minutes shaking, in order to form butter of the fat. Then the ether is added and the process continued as usual. In this case the percentage obtained by the areometric method must be increased .1 per cent. in order to agree with the gravimetric determinations. Schmoeger further recommends that skimmed milk or sweet buttermilk after treatment with potash be shaken with 10 grams potassium sulphate until the latter is dissolved. But this method also influences the specific gravity of the ether-fat solution, and the corrections to be made are found in the table given.²

Soxhlet himself³ has called attention to the fact that with skimmed milk the ether-fat solution does not readily separate. A special scale has been constructed for such fat-poor milks giving areometric readings from 21.1 to 43, with the corresponding percentages of fat. For such milks Soxhlet proposes the following treatment, viz: A soap solution is made by taking 15 grams of a stearine candle, adding to it 25cc. alcohol, and 10cc. of the potash solution of the strength before given. The stearine is saponified by heating the mixture, and after the solution has become clear it is made up to 100cc. with water. From .4 to .5cc. of this solution is added to the milk under examination, and after a good shaking the rest of the process is carried on in the usual way. After the first thorough shaking on the addition of the ether the light jolting must be continued for fifteen minutes at intervals of half a minute in order to have the ether solution collect at the top. At longest, the solution separated after three or four hours.

Halenke and Möslinger⁴ call attention to the fact that if samples of milk are kept for some time, even on ice, the ether-fat solution will no longer separate. They prefer in such cases a modification of Liebermann's method, which they describe. In general I may say the areometric method has met with the approval of all analysts who have used it with exception of Preusse⁵, but Soxhlet⁶ has shown that Preusse did not understand how to use the apparatus.

¹ *Op. cit.*

² *Ibid.*, p. 132.

³ Zeit. landw. Ver. Bayern, 1882, p. 18.

⁴ Ver. Bay. Vertreter d. Angewand. Chem., p. 110.

⁵ Mittheil. Reichsgesundheitsamt, vol. I, p. 378.

⁶ Zeit. landw. Ver. Bayern, 1881, p. 700.

The following chemists, in addition to those already mentioned, have given the method their entire approval: Egger, Kellner, Schrodtt, Friedländer, Meissel, Fleischman, Hofmeister, Deitzell, Moser, Schreiner, Janke, Gerner, and Angström.

I will give now some of my own experiences with the areometric method:

The milk examined by me was mostly obtained from a neighboring dairy and was a mixture from forty cows. Samples were also bought from dealers in the city. The milk from the dairy mentioned was drawn at 5 p. m., and the examination made the following morning. This may partially account for the small success I had in securing a good separation of the ether-fat. The work extended from March 23 to May 7, 1886.

With the first series of samples in which the method of separation recommended by Soxhlet was followed ninety-three trials were made. In only four cases was the separation sufficiently good to get a reading within thirty minutes. A larger number of readings was obtained within an hour, and about half the number could be read at the end of three or four hours. Of the remainder about one-half could be read after twenty-four hours, and the rest did not separate at all. The results of reading the areometer at different times, however, showed that the density of the ether-fat solution underwent quite a change. The following data will show the nature and extent of this change:

No.	Per cent. fat.		
	First results.	Results after 24 hours.	Difference.
1.....	4.16	4.08	-0.08
2.....	3.52	3.83	0.31
3.....	3.20	3.52	0.30
4.....	3.63	3.68	0.05
5.....	5.28	5.28	0.00
6.....	4.81	5.13	0.32
7.....	4.61	4.71	-0.10

From the above it is seen that there is no uniformity in the character of this change, but in the greater number of cases the areometer shows an increase in the percentage of fat on standing.

Attempts also to obtain a more perfect separation by varying the quantity of potash employed gave only conflicting results.

I was, therefore, forced to the conclusion that for general work Soxhlet's method would prove useless unless some method could be devised to secure a prompt and uniform separation of the ether-fat solution.

Various theories have been proposed to account for this peculiarity of milk in refusing to allow the ether solution to separate. Caldwell and Parr have supposed it to be due to the bran in the cow's food; Liebermann ascribes it to failure of manipulation; Schmoeger that it is caused

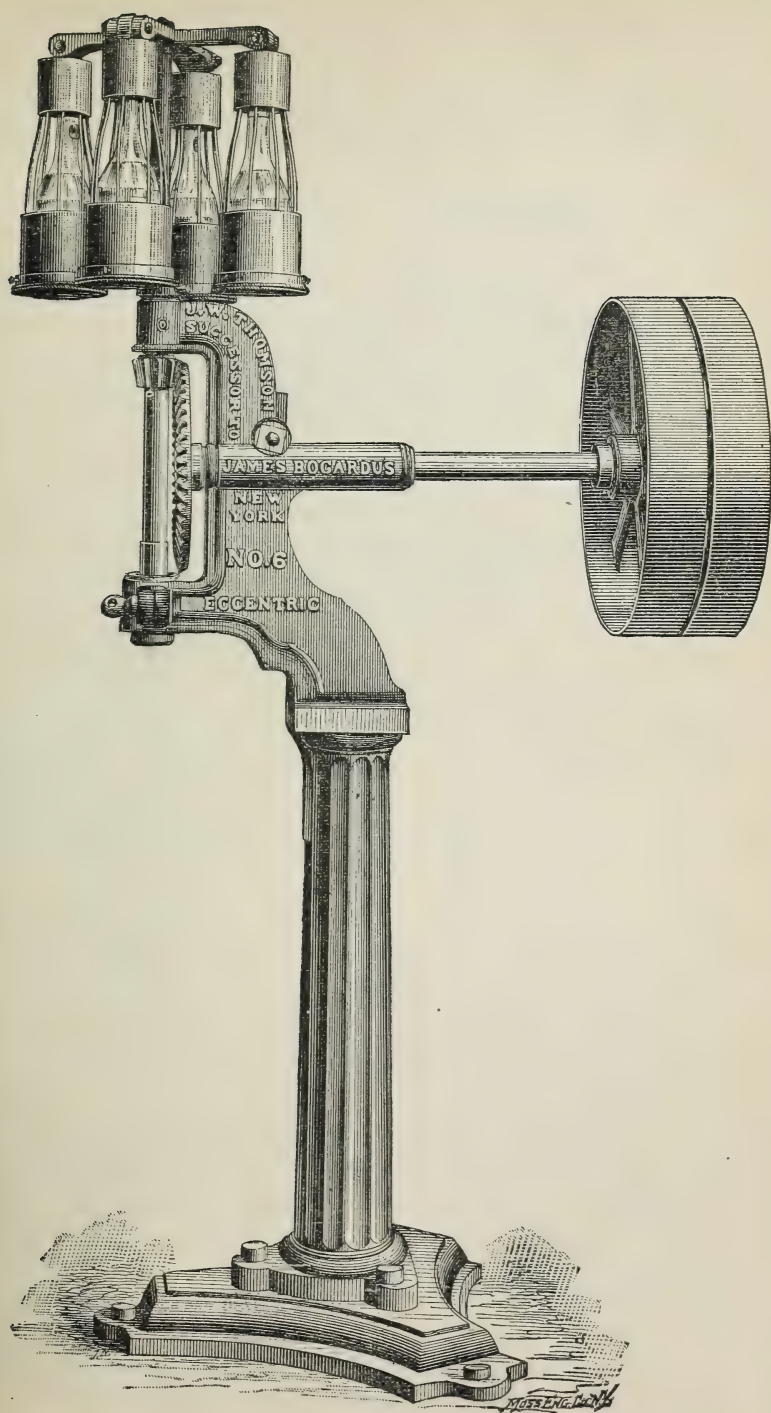


FIG. 2.

by the milk standing on ice; Soxhlet thinks it is the result of deficiency of fat; and others attribute it to differences in age and breed of the cows. The résumé which precedes shows that not only the actual volume of the ethereal solution, but also the time of the separation required, has a serious disturbing influence on the specific gravity of the ether-fat solution.

Therefore, the method, in order to be of general application, must be subjected to some radical modification.

In this direction were the attempts to secure a more prompt separation by varying the amounts of caustic-potash solution employed. These attempts, as the record has shown, were entirely unsuccessful. Even if the different kinds of milk would permit a prompt separation by varying the quantities of alkali employed, the amount for each sample could only be determined by numerous and tedious experiments.

I, therefore, turned my attention in another direction. It seemed to me that a centrifugal machine might be used to secure this separation, and accordingly I had a castaway drug-mill, formerly used in the laboratory, modified so as to serve for this purpose. The machine was so arranged as to hold four separatory flasks and impart to them a high speed of rotation. The form of the machine, with modifications made, is shown in figure 2.

At this point of my investigations this apparatus was finished and I immediately subjected it to a trial.¹

Four samples which had not separated at all at the end of three hours were placed in the apparatus and whirled for ten minutes. At the end of this time three of them had completely separated, and the fourth nearly so. The apparatus was set in motion again for five minutes, at the end of which time the separation of the fourth sample was accomplished.

The number of revolutions per minute of the machine was about 350.

It will be seen from the above that the very first trial of the machine was completely successful, securing a perfect separation of the ether-fat solution in a few moments in samples which previous trial, by the usual method, had failed to separate in several hours.

The next determinations were made on a sample of milk purchased at the Department restaurant.

Duplicate flasks were treated in the usual way to secure the separation, and only at the end of two and a half hours was enough clear solution obtained to get a reading: No. 1 gave 2.40 per cent. fat; No. 2, 2.30 per cent. fat.

The first set of samples of the same milk separated by the centrifugal gave the percentages following: No. 1, 5.52 per cent. fat; No. 2, 2.32 per cent. fat.

¹This apparatus was first described before the Chemical Society of Washington, May, 1886, and next at the Buffalo meeting of the A. A. A. S., August, 1886.

The separation took place perfectly in ten minutes, with a rate of revolution of about 300 per minute.

The second set of four samples was treated in the same way and separated completely in eight minutes. The following readings were obtained: No. 1 gave 2.36 per cent. fat; No. 2, 2.34 per cent. fat; No. 3, 2.31 per cent. fat; No. 4, 2.30 per cent. fat.

The third set of samples separated by the centrifugal showed the following percentages: No. 1 gave 2.23 per cent. fat; No. 2, 2.30 per cent. fat.

The volume of the clear ether-fat solution in each case was about 40cc.

The next trial was with milk also purchased in the Department restaurant. It proved to be one of the rare cases in which a reasonably prompt separation was secured by the old method. After thirty minutes about 25cc. of the ether solution had separated, which was enough to get a reading. Duplicate determinations were made: No. 1 gave 2.08 per cent. fat; No. 2, 2.04 per cent. fat.

Four separations of the same milk were also made with the centrifugal. Separation took place promptly in eight minutes at a speed of about 200 revolutions per minute, and the volume of ether-fat in each case was about 40cc.: No. 1 gave 2.01 per cent. fat; No. 2, 2.01 per cent. fat; No. 3, 2.00 per cent. fat; No. 4, 2.04 per cent. fat; which is an agreement as close as any one could expect.

Having thus shown that the centrifugal method was capable of making the areometric method applicable to almost every sample of milk, I undertook a new series of experiments. In all, 155 samples were subjected to treatment.

Of the 155 samples examined only 57 gave a good separation by the Soxhlet method in thirty minutes. Of the remaining 98, about half did not separate at all so as to permit a reading, and the other half only after several hours. Compare this with the centrifugal method, in which only 6 samples out of the whole lot required over fifteen minutes for separation and only one was abandoned as entirely inseparable, and the more general application of the process is at once apparent.

Of the 6 samples mentioned above, 3 were from the same cow, a grade Shorthorn, four years old, weight about 800 pounds, in milk since July 1, 1885. She gave 6 quarts of milk a day, was milked at 5 a. m. and 5 p. m. The samples of milk sent were taken at 5 p. m., on April 13, 17, and 22, respectively. The food received by this cow was the same as for all the others (36) from which samples were taken for analysis. They received at 5 a. m. 3 pounds of wheat bran, and the same of hominy chops, and then as much corn (maize) fodder as they could eat. The bran and chops were fed dry. In pleasant weather the cows were out until 3 p. m. They were then fed 10 pounds each of unthrashed oats. At 5 p. m. they got a half peck of chopped turnips and a repetition of the morning's feed of bran and chops.

The hominy chops used showed, on analysis, the following composition :

	Per cent.
Water	7.13
Ash	2.53
Ether extracts	9.03
Carbohydrates	69.32
Crude fiber	2.36
Albuminoids	9.63

Two of the other samples were received April 27 and 30 from a thoroughbred Jersey, four years old, weight about 600 pounds, in milk since July 1, 1885, giving at the time about 5 quarts daily. On the 29th of April samples of milk were also treated from the same cow, but after dilution the centrifugal separation, although more than usually difficult, did not require so long a time as on the occasion mentioned.

There is nothing shown by the analysis, by the breed of cow, nor by the food which gives any definite idea of the cause of the peculiarity in these milks which does not permit a speedy separation. It certainly is not the quantity of fat present, for other milks having the same, more or less, amounts of fat separated without difficulty. In the absence of any further evidence on this point we can only attribute the phenomenon to bovine idiosyncrasy.

In all 90 samples were compared by the usual method of separation and by the centrifugal. By the former method the mean percentage of fat obtained was 4.01 and by the latter 3.88. It thus appears that the numbers obtained by the centrifugal method must be increased by .13 in order to correspond to those of the old method. This discrepancy is readily explained when it is remembered that by the centrifugal motion the percentage of ether left in emulsion would naturally be less than with the former process of separation. The ether-fat solution thus becomes more dilute and consequently has a lower specific gravity. When, therefore, the percentage of fat in a milk determined areometrically, is calculated by the tables given for the old method of separation, it should be increased by .13 in order to represent the actual quantity present.

I think it safe to conclude from the data which have been obtained :

First, that the method of Soxhlet cannot be applied to the determination of fat in American milks, especially if they be from individual animals. It works somewhat better on mixed milks from a large dairy, but even in this case it is a rare thing to secure a prompt separation and in most cases the method would be very difficult of application.

Second, that by the use of the centrifugal machine described a prompt separation of the ether-fat solution can be obtained in all cases, even in those in which after forty-eight hours no separation whatever takes place by the usual method.

Third, that the estimation of the fat in milk by Soxhlet's areometer can only be accurately secured when standard volumes of aqueous ether

and caustic potash are employed, when the volume of the ether-fat solution separated is sensibly constant and the time employed in separation sensibly the same. These conditions can only be secured by the use of the centrifugal machine described.

I propose to use a centrifugal apparatus also for assisting in the separation of the ether-fat solution in the lactobutyrometer; and it has already proved its usefulness in separating precipitates which subside very slowly.

I am of the opinion that such a machine would prove of great value in every chemical laboratory aside from its utility in determining the fats in milk.

Cronander¹ has proposed the following method of estimating the fat in milk:

A glass flask of 200 to 250cc. capacity, and two glass tubes constitute the chief parts of the apparatus. One of the tubes is furnished with a scale dividing it into ten equal parts. Below the last division the scale is expanded into a bulb, below which the tube extends for about 5cm. The other tube is bent to an obtuse angle and serves for the introduction of hot water into the flask to drive the fat into the measuring tube at the end of the operation. Both tubes are fastened to a cork stopper in such a manner as to have the measuring-tube end even with the under surface of the stopper, while the other extends almost to the bottom of the flask.

Of the milk to be analyzed 100cc. are taken at 17.5° C., 10cc. potash lye (200 grams to the litre) added and 30cc. aqueous ether. The flask is corked and thoroughly shaken. The ether-fat solution collects at the top (after one hour), and after evaporating the ether the residual fat is forced into the measuring tube by pouring water at 70° C. to 80° C. into the flask. The volume of the fat is thus determined and its per cent. can be calculated.

LIEBERMANN'S METHOD.²

This method, like that of Soxhlet's, depends on the separation of a fat from a mixture of milk and caustic potash by shaking it with ether.

Apparatus.—(1) A glass cylinder with ground glass-stopper, 26cm. high and 3.5cm. diameter. (2) Burettes of the form shown in Figs. 3, 4. (3) A glass flask holding from 45 to 47cc., according to size of burette employed; neck 1cm. diameter, with edge ground accurately in a horizontal plane. (4) Four pipettes, two of 50cc. and one each of 20cc. and 5cc. Before beginning the operation the flask is graduated as follows:

The burette, Fig. 3, is filled to the zero-point with pure water at the temperature of the working room. The water is now run out of the

¹ Milchzeitung, vol. 11, pp. 161-164.

² Zeit. Anal. Chem., 1883, p. 383; 1884, p. 476; 1884, p. 87.

burette into the flask (previously carefully cleaned and dried), until the meniscus at the ground edge of the neck passes from the concave to the convex form, a change which is effected by a single drop. When

the flask is nearly full at least five minutes should be allowed for the water to settle in the burette. The cubical contents of the flask are now noted from the burette.

Reagents.—A solution of caustic potash having a specific gravity of 1.27. A solution of aqueous ether the same as is used in Soxhlet's method.

Manipulation.—Fifty cubic centimeters of the milk in the cylinder described above are treated with 5cc. of the potash solution, well shaken and allowed to stand for five minutes. This is next treated with 50cc. of the aqueous ether, and gently shaken for ten seconds. The cylinder is now allowed to stand for twenty minutes, receiving every half minute one or two light vertical blows. At the end of this time the separation of the clear ether layer is usually complete, yet it sometimes happens that some of the emulsion adheres to the under part of the flask in the form of a transparent covering. By means of a gentle rotating movement imparted to the cylinder this emulsion is collected and rapidly settles.

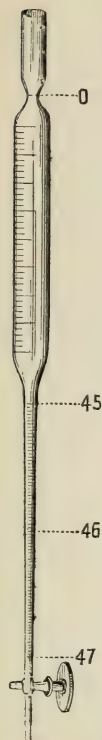


FIG. 3.

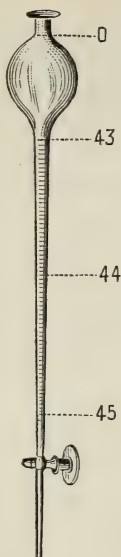


FIG. 4.

With a 20cc. pipette the clear ether solution is removed. Before this is allowed to flow into the flask that part of it which has been dipped into the solution is carefully wiped, so that no part of the potash liquid can drop into the flask. The ether solution is now evaporated, and the residue dried for at least half an hour at a temperature of $110^{\circ}\text{C}.$, or what is still better, over a small flame until the odor of the decomposed butter is detected. The whole is now cooled in a dessiccator. It can now be weighed, if the weight of the flask is known, or it can be estimated volumetrically.

In case the estimation is made volumetrically, it must be carefully observed that the solidified fat contains no air bubbles. In case any air bubbles are noticed the fat must be again melted and warmed until they have all disappeared.

Into the flask containing the solidified butter fat water is again run out of the burette under the same conditions which obtained in determining the contents of the flask at first. It is of the greatest impor-

tance that all the measurements be made with the greatest exactitude, since a single drop too much or too little will influence the result.

In case some small particles of the butter fat are detached and swim about in the liquid no fear need be entertained that the results of the measurement will be influenced thereby.

It will appear at once that the difference between the volume of water originally held by the flask, and that which was necessary to fill it after the fat had been added, will represent the volume of the fat which was contained in the 20cc. of the ether solution. This number multiplied by 5 will give the volume per cent. of the fat at the temperature at which the experiment was made.

For converting volume per cent. into weight per cent. the following table is used:

Table for converting volume per cent. into weight per cent.

At temperature 15° C., volume per cent. X .91109 = weight per cent.	
16°	.90831
17°	.90642
18°	.90377
19°	.90170
20°	.90034
21°	.89837
22°	.89626
23°	.89216
24°	.88822
25°	.88703
26°	.87584
27°	.87463
28°	.87327
29°	.87191
30°	.87055

The following example will show the manner in which the above-described method of analysis may be reckoned.

The graduation of the flask shows a volume of 48cc. and 25cc. After the evaporation of the 20cc. of the ether solution 47.3cc. water was necessary to fill the flask. The volume of fat which was contained in the 20cc. of the ether solution is therefore $48.25 - 47.38 = .95$ cc. This number multiplied by 5 gives 4.75cc. volume per cent. of fat. The temperature at which the estimation was made was 17° C.

Looking now in the table, opposite 17° C. we find the factor .90642 This number multiplied by 4.75 gives 4.3, which is equal to the per cent of fat by weight.

If it is wished to determine the per cent. by weight of butter per 100 grams, and not cubic centimeters as before, it is necessary to determine the specific gravity of the milk and to proceed according to the following formula:

$$P = \frac{p}{S} \frac{1000}{S}$$

In this formula P denotes the number sought, p the quantity of fat found for 100 cubic centimeters, and S the specific gravity of the milk.

According to Wolff the percentage of fat obtained by the method just mentioned is too high, because a part of the ether used for separat-

ing the fat remains dissolved in the alkaline milk and this part of the ether contains no fat. The result is that the ethereal solution of the fat contains more of this substance than it otherwise would if the whole of the ether was separated.

Wolff has therefore proposed the following changes in the quantities of the reagents to be used and claims thereby to have obtained results which wholly agree with the estimations of fat by weight. For 50cc. of milk he proposes 3cc. of potash lye of 1.145 specific gravity and 54cc. of the aqueous ether.

Liebermann, however, in a review of the methods proposed by Wolff, maintains that his original method gives entirely reliable results.

FLEISCHMAN AND MORGEN'S METHOD.²

Fleischman and Morgen describe a method of determining fat in milk when the specific gravity and total solids are known.

Fleischman³ gives a more detailed study of this method.

The formulæ for the calculations are as follows:

$$(1) \quad t = 1.2 f 2,665 \frac{100S - 100}{S}$$

$$(2) \quad f = 0.833 - 2.22 \frac{100S - 100}{S}$$

In these formulæ t =per cent. total solids; f =per cent. fat in milk; S =specific gravity of the milk at 15° C.

The above formulæ may be simplified by putting

$$d = 100S - 100.$$

Tables are given to aid in the calculation of the results. By these formulæ when either the per cent. of fat or the total solids is known the other can be calculated with a high degree of accuracy.

ESTIMATION BY VOLUME OF CREAM.

The determination of the volume of cream gives a rough approximation of the percentage of fat in the milk.

The methods generally in use are based on the natural separation of the fat globules on standing and the estimation of the volume thereof in a graduated cylinder.

The creamometer of Chevalier⁴ will serve as a type of all apparatus of this class. It is a cylinder 20cm. high and 40cm. diameter. The scale

¹ Pharm. Centralh., vol. 24, p. 435; Zeit. Anal. Chem., 1884, p. 87.

² Jour. Landw., 1882, pp. 293-301.

³ Jour. Landw., 1885, p. 251 *et seq.*

⁴ Becke, Milchprüfungs-Methoden, p. 40.

begins at 5cm. from the top of the cylinder and is extended downwards. Each mark is one-hundredth of the whole volume. Being filled with milk to the zero point and allowed to stand twenty-four to forty-eight hours, the percentage of cream is read directly on the scale.

Since the volume of cream formed depends on the shape of the vessel the temperature, and the time, this method is not reliable. This error is reduced to a minimum by the use of a centrifugal machine for separating the cream. A machine for this purpose has been constructed by Lefeldt.¹

In 1883 I saw a very convenient machine which had been constructed, in the laboratory of the University of Illinois at Champaign. The apparatus already described for separating the ether fat solution in Soxhlet's method I have used with success in separating cream. The centrifugals used in separating the cream from the milk in large dairies are constructed on the same principle.

For a comparison of the numbers obtained by these processes with those given by the gravimetric determination, I refer to Becke's monograph.²

The Lactocrite.—This name is given to an instrument invented by De Laval³ designed to separate the fat in milk after appropriate chemical treatment.

The test vessels used in this apparatus are cylindrical boxes made of silvered metal, with accurately ground hollow silvered stoppers. These stoppers are expanded at the bottom, and at the top are contracted, and end in a small hole. They are joined to a glass tube of small internal diameter. This tube is furnished with a jacket by which it can be screwed onto the stopper, and this jacket carries two longitudinal slits, through which the divisions in the glass tube can be read. In the bottom of the jacket is a hole, so that the glass tube and metal stopper form a canal open at both ends.

The centrifugal machine consists of a steel revolving disk. In the upper part of this there is a circular cavity, extending from which, like radii, are 12 holes to receive the test apparatus above described. These holes dip slightly downward. The disk is incased with a jacket having a removable cover, which prevents a too rapid fall of temperature during the operation. By means of appropriate apparatus the disk can be driven at the rate of about 6,000 revolutions per minute.

Preparation of the milk.—In an ordinary test tube put equal portions of the milk to be tested and a mixture of 20 parts of concentrated acetic and one part of sulphuric acid. The test tube is closed with a cork in which is fixed a glass tube, shaken, and heated for ten to fifteen minutes in a water bath with frequent shaking.

¹ Becke, *op. cit.*, p. 43.

² *Op. cit.*, pp. 40-45.

³ Ding. Poly. J., vol. 261, p. 219; Chem. Centralblatt, 1886, p. 798.

The cylindrical box above described is now filled from the test tube, the metal cork forced in, whereby the apparatus is entirely filled and the excess of milk forced out through the holes in the bottom of the jacket.

The disk having been warmed to 50° to 60° C. by hot water, is now filled with these samples and revolved for three to five minutes at the velocity already noted. The temperature of the disk should not be allowed to fall below 50° C.

At the end of this time the fat has completely separated and its volume can be read on the divisions of the glass tube. This division is so arranged as to represent .1 per cent.

Blyth¹ has made a comparison of the results furnished by the lactocrite with those obtained by Adams method. The results are given in the following table:

No.	Specific gravity.	Total solids.	Ash.	Fat.		
				Lactocrite.	Adams.	Difference.
1	1033.0	12.90	.80	3.40	3.44	-.04
2	1031.0	14.12	.80	4.55	4.69	-.14
3	1032.0	13.12	.77	3.60	3.57	.03
4	1030.5	13.07	.74	3.90	3.99	-.09
5	1032.0	12.98	.76	3.80	3.76	.04
6	1031.5	14.27	.76	4.90	4.84	.06
7	1032.5	13.84	.78	4.20	4.26	.06
8	1030.5	13.00	.76	3.70	3.69	.01
9	1031.0	13.51	.80	4.05	4.09	.04
10	1034.0	11.76	.76	2.10	2.07	.03
11	1035.0	9.99	.86	.45	.50	-.05

The table shows the greatest differences between the two estimations to be .14 and the mean difference to be .05 per cent.

It is estimated that with the lactocrite 48 determinations of fat in milk can be made in an hour.

A further discussion of the merits of the lactocrite is given by Faber.² He says:

One great advantage of the lactocrite is the very simple way in which it is worked, so that no skill is necessary, but any dairyman may obtain as good results as the apparatus is able to yield. In order to illustrate this, I give below the results obtained by two persons at their first attempts; the first person is a dairyman used to heavy work. By way of a check I myself made some tests of the same milks:

By myself.	Dairyman.		
3.1	3.1	3.2	3.2
3.2	Failed	3.2	3.2
3.2	3.1	3.3	3.2
2.65	2.65	2.6	2.6
2.65	2.65	2.6	2.65

These very favorable results are of importance as showing that in the lactocrite is at last found the long wished-for apparatus, possessing the two qualities not hither-

¹Analyst, 1887, p. 34.

²Analyst, 1887, pp. 6 *et seq.*

to combined—simplicity of construction and working and sufficient correctness for all practical purposes.

The lactocrite will, no doubt, be found invaluable for butter dairies, or dairy factories buying milk from different farmers, by enabling them to carry out the system of paying for the milk according to the amount of butter-fat which is the only fair system. At present, both in England and in other countries, the farmer whose milk will make butter at a rate of 3 pounds per 100 pounds of milk gets the same price as the farmers whose milk is so rich as to give 5 pounds of butter per 100 pounds of milk, which of course is most unfair. When milk is paid for according to the fat contained in it, the temptation to skim it is done away with, and, besides, a great encouragement is given to the production of rich milk.

The lactocrite will also prove of use for analysts who have access to a separator stand, as it will give in short time a more exact determination of the amount of fat than any other apparatus. In this connection it will be of interest to know that a special construction of it has been adapted to fit Dr. De Laval's small hand separator, worked by hand and requiring no foundation.

Sebelien has published a comparison of the results obtained with the lactocrite and Cronander's method with the gravimetric methods.¹

Cronander's method gave in general results slightly below those furnished by the gravimetric and Soxhlet's processes.

Dr. Cronander, to avoid this error, has introduced a slight modification into his process by adding a little alcohol to the mixture of potash, ether and milk. The principle of the separation of the fat thus becomes the same as in the lactobutyrometer of M. Chevreul. By using this modified process it was found possible to bring the results up near to those of the gravimetric method.

The results furnished by the lactocrite showed an almost perfect agreement with the gravimetric numbers, the differences being usually within 0.05 per cent.

Attention must be paid to keeping the test tube holding the milk and acids well shaken, especially before pouring its contents into the metal box, and that the rest of the apparatus be pressed in the box at once when the milk has been found. In proceeding in this way no separation of the different parts of the test liquid is possible, and thus a fair average sample is recovered in the test glass.

Concerning the question of the advantages of the lactocrite as compared with other forms of apparatus for estimating the fat in milk Sebelien is somewhat conservative, but seems to think that the matter will soon be determined by comparative trials.

LACTOBUTYROMETRIC METHOD.²

This volumetric method depends on the separation of the fat from the milk by a mixture of ether and alcohol. The method has been carefully studied by Caldwell and Parr.³

A mixture of 75 parts of pure ether, 100 of absolute alcohol, and 135 of water are employed. The instrument employed is made of moderately thick-walled tubing (about

¹ Landw. Versuchs-Stationen, vol. 33, pp. 393 *et seq.*

² Marchand, Instruction sur l'emploi du lactobutyrometer, Paris, 1856 and 1878.

³ Am. Chem. Jour., vol. 7, pp. 238 *et seq.*

1mm.); the stem is about 23cm., and the bulb about 8cm. long. It is important that the shoulder between the stem and the bulb should not be too abrupt. The bore of the stem is about 6mm., and it is graduated in $\frac{1}{2}$ cc. The wider part of the tube has such a capacity that in passing from the lowest graduation on the stem to the inner end of the stopper in the lower mouth one passes from 5 to 33cc.; then the ether-fat solution will always come within the range of the graduation on the stem. This instrument differs from that originally given by Marchand only in being open at the bottom as well as at the top; this is a matter of some importance with reference to cleaning and drying it. The narrow stem in which the ether-fat solution collects makes more accurate readings possible than is the case with the wider tube with the same width of bore throughout, such as is now commonly used.

The manipulation is carried on as follows :

Closing the lower mouth with a good cork, 10cc. of the well-mixed sample of milk are delivered into the well-dried tube from a pipette, then 8cc. of ether (Squibbs's stronger) and 2cc. of 80 per cent. alcohol. Close the smaller mouth of the tube with a cork, and mix the liquids by thorough shaking, which, however, need not be either violent or prolonged. Both corks should be held in place by the fingers during this operation, and the upper one should be once or twice carefully removed to relieve the pressure within, otherwise it is liable to be forced out suddenly unless carefully watched, with consequent danger of loss of material. Lay the tube on its side for a few minutes and then shake it again, add 1cc. of ordinary ammonia diluted with about its volume of water, and mix as before by shaking; then add 10cc. of 80 per cent. alcohol, and mix again thoroughly by moderate shaking, and holding the tube from time to time in an inverted position while the lighter portion of the liquid rises to the surface.

Now put the tube in water kept at 40° to 45° C. till the ether-fat solution separates; this separation may be hastened by transferring the tube to cold water after it has stood in the warm water for a few minutes and then returning it to the warm water. Finally transfer the tube to water kept at about 20° C., and as the level of the liquid falls in the stem by the contraction of the main body of it in the bulb, gently tap the side of the tube below the ether-fat solution, to dislodge any flakes of solid matter that may adhere to the walls; then as this solution finally takes its permanent position in the tube, its volume will not be increased by the presence of such foreign matters. The readings are to be taken from the lowest part of the surface meniscus to the line of separation between the ether-fat solution and the liquid below it.

In this laboratory the use of the lactobutyrometer has been attended with the same difficulties, though to a less extent, which led to the modification of Soxhlet's method already noticed. The late improvements in both the volumetric and gravimetric determinations of fat in milk render a further discussion of the merits of the lactobutyrometer unnecessary.

OPTICAL METHODS OF ESTIMATING FAT IN MILK.

Since the white color of milk is due to the suspension of the fat globules, many devices have been contrived to determine the quantity of fat present by the opacity of the milk. The most convenient of these apparatus is the one designed by Feser.

It consists of a glass cylinder, in the lower part of which a smaller cylinder made of white glass is fixed. On this white glass are a few black lines. The outer cylinder carries a double scale, one set of numbers representing cubic centimeters and the other the percentage of fat.

Four cubic centimeters of milk are put in the cylinder and then water added until the black lines on the inner white cylinder become visible. The percentage of fat is then read from the top of the column of water in the large cylinder.

For a full description of the different forms of lactoscope the monograph of von der Becke may be consulted.¹ For sorting milks, the lactoscope in the hands of an experienced operator will give valuable indications in respect of the quantity of fat. A delicate lactometer, a good lactoscope, and an experienced operator will generally be able to determine whether a given sample of milk be whole or skimmed. The lactoscope, however, is of no value in determining with accuracy the percentage of fat present in a sample of milk.

ESTIMATION OF LACTOSE.

Chemical.—The chemical methods employed in estimating the sugar in milk will be fully discussed in another part of this bulletin devoted to the study of sugars and their adulterations.

Optical.—The optical method of determining the quantity of lactose in milk is both speedy and accurate when properly carried out. The principles which underlie this investigation and the proper method of carrying it out are given below.²

The usual method of determining milk sugar by evaporating the sample to dryness and extracting the sugar with alcohol after exhausting with ether requires a great deal of time and labor. If some reliable optical method could be devised the determination of the lactose in milk would be the work of only a few minutes. The difficulties which are encountered in seeking for such a method are numerous and serious, so much so that little credit has heretofore been given to any of the processes of optical analysis in use.

SPECIFIC ROTATORY POWER OF MILK SUGAR.

Crystallized milk sugar when first dissolved possesses a higher rotatory power than it has in the milk from which it was derived. This increased optical activity may be compared with the original by the ratio 8:5, nearly. After the solution has stood for twelve to twenty hours, or immediately on boiling it, this extra rotatory power is lost. In estimating the specific rotatory power of milk sugar the numbers given always refer to the constant and not the transient gyrotory property.

Among the earliest numbers assigned to the rotation of lactose are those of Poggiale (a)_v = 54.2 and Erdmann (a)_v = 51.5 [Sucrose (a)_v = 66.5]. Biot³ places this number for lactose at 60.23, and Berthelot⁴ at 59.3 for the transition tint (a)_j. Hoppe-Seyler, in his "Handbuch der physiologisch-chemischen Analyse," gives this number at (a)_j = 58.2. Since the

¹ *Op. cit.*, pp. 45 *et seq.*

² *Am. Chem. Jour.*, vol. 6, pp. 289 *et seq.*

³ *Compt. Rend.*, vol. 42, p. 349.

⁴ *Wüirtz Diet. de Chim.*, vol. 2, 1st part, p. 188.

ratio of $(a)_o$ to $(a)_j$ is 1 : 1.1306, the above numbers become for Biot $(a)_o = 53.27$, for Berthelot $(a)_o = 52.47$, and Hoppe-Seyler $(a)_o = 51.48$. Hesse¹ observed the rotation number to be $(a)_o = 52.67$ when the solution contained 12 grams per 100cc. and the temperature was 15° C. On the other hand, when the concentration is only 2 grams per 100cc. the number assigned is $(a)_o = 53.63$. It appears from this that the specific rotation power of a solution of milk sugar diminishes with the increase of its concentration, and this view is adopted by Landolt, Tollens, and Schmidt.

The following general formula² is used to correct the reading of the polariscope for concentration of solution :

$$(a)_o = 54.54 - .5575c + .05475c^2 - .001774c^3,$$

in which c = number grams sugar in 100cc. solution. These observations are contradicted by the work of Schmoeger,³ who, in an elaborate series of experiments, using instruments of different construction and observing all necessary precautions, found the rotation number of lactose sensibly constant for all degrees of concentration up to the saturation point. In thirty-two series of investigations, in which the degree of concentration gradually increases from $c = 2.3554$ to $c = 36.0776$, and in which a constant temperature of 20° C. was maintained, the variations in the numbers obtained were always within the limits of error of observation. The mean of all these numbers fixes the value of $(a)_o$ at 52.13.

According to Schmoeger variations in temperature have far more to do with changes in rotatory power than differences of concentration. The value of $(a)_o$ falls as the temperature rises. Under 20° C. the disturbing influence of temperature is greater than above 20° C. At the latter degree $(a)_o$ varies inversely about .075 for each 1° C. change of temperature. Pellet and Biard,⁴ as a result of their observations, fix the rotatory power of milk sugar at 53.94 for $(a)_j$ [δ . $(a)_o = 52.12$].

After a careful review of the methods used in the above *résumé* and the numbers determined by them, I am inclined to accept the mean obtained by Schmoeger as the one entitled to the greatest credit. It also has the advantage of being almost the mean of all the various numbers which have been assigned as the specific rotating power of lactose, viz :

Poggiale.....	54.20
Erdmann	51.50
Biot	53.27
Berthelot	52.47
Hoppe-Seyler	51.48
Hesse.....	52.67
Hesse.....	53.63
Schmoeger.....	52.53
Pellet and Biard	52.12
Mean.....	52.65

¹Anal. Chem. u. Pharm., vol. 176, p. 98.

⁴Bull. de l'Assoc. des Chimistes vol. 1, p. 171 *et seq.*

²Tucker, Sugar Analysis, p. 91.

³Ber. chem. Gessell., vol. 12, p. 1922 *et seq.*

In the present state of our knowledge, therefore, the specific rotatory power of milk sugar should be taken at $(a)_D = 52.5$. I propose, at an early date, to make a careful study of this subject, in order to fix, if possible, an exact number for the expression of the rotating power, and to examine the conflicting evidence respecting the influence of the degree of concentration on the same. The estimation of lactose in milk by the polariscope is rendered difficult also by the presence in milk of various albumens—all of which turn the plane of polarization to the left. As will be seen by the data given further along, the ordinary method of removing these albumens, viz, by a solution of basic lead acetate, is far from being perfect. If, therefore, a portion of the albumen be left in the liquid submitted to polarization, the rotation to the right will be diminished by its presence.

Hoppe-Seyler¹ assigns as the rotation power of egg albumen $(a)_D = -35.5$, and for serum albumen $(a)_D = -56$. Both acids and alkalis seem to increase the rotating power, which may with acetic acid reach $(a)_D = -71$.

Fredericq² gives the rotation number for blood serum for the rabbit, cow, and horse at $(a)_D = -57.3$, and for the dog at -44 . Paraglobulin, according to the same author, has a rotation number $(a)_D = -47.8$.

Milk albumen³ has the following numbers assigned to it:

Dissolved in Mg.SO ₄ sol.	$(a)_D = -80$
Dissolved in dil. HCl.	$(a)_D = -87$
Dissolved in dil. NaOH sol.	$(a)_D = -76$
Dissolved in strong KOH sol.	$(a)_D = -91$

The hydrates of albumen⁴ have rotation powers which vary from $(a)_D = -71.40$ to $(a)_D = -79.05$. From the chaotic state of knowledge concerning the specific rotating power of the various albumens, it is impossible to assign any number which will bear the test of criticism. For the purposes of this report, however, this number may be fixed at $(a)_D = -70$ for the albumens which remain in solution in the liquids polarized for milk sugar.

The phenomenon of "birotation" in milk sugar has already been noticed. The problem of analysis of this sugar is, however, still further complicated by the facts pointed out by Schmoeger⁵ and Erdmann,⁶ that when milk is rapidly evaporated in a plain dish the sugar is left in the anhydrous state, and that this sugar in fresh solutions exhibits the phenomenon of "half rotation." When such sugar is extracted with alcohol and re-evaporated, it, doubtless, is still anhydrous. But in the calculation of results this sugar is generally estimated as containing water of crystallization, and thus an error, which Schmoeger reckons at as much as .2 per cent., is introduced into the results. This

¹ Würtz, *Diet. de Chimie*, vol. 1, 1st part, p. 91.

² *Compt. Rend.*, vol. 93, p. 465.

³ Hoppe-Seyler in *Handbook of the Polariscope*, Landolt, p. 248.

⁴ Kühne and Chittenden, *Am. Chem. Jour.* vol. 6, p. 45.

⁵ *Ber. chem. Gesell.*, vol. 12, 1915 *et seq.*; vol. 13, p. 212 *et seq.*

⁶ *Ber. chem. Gesell.*, vol. 12, p. 2180 *et seq.*

fact, not well recognized, combined with the knowledge that in the process of evaporation many particles of sugar must be occluded by the hardening caseine, tends to throw doubt upon the accuracy of estimating the sugar by the extraction method.

The work which I undertook had for its object the determination of the best method of preparing the milk-sugar solution for the polariscope, and a comparison of the numbers obtained by this instrument with those given by the ordinary process of extraction.

The reagents used for removing the albumens were:

- (1) Saturated solution basic lead acetate, specific gravity 1.97.
- (2) Nitric acid solution of mercuric nitrate diluted with an equal volume of water.
- (3) Acetic acid, specific gravity 1.040, containing 29 per cent. $\text{HC}_2\text{H}_3\text{O}_2$.
- (4) Nitric acid, specific gravity 1.197, containing 30 per cent. HNO_3 .
- (5) Sulphuric acid, specific gravity 1.255, containing 31 per cent. H_2SO_4 .
- (6) Saturated solution sodium chloride.
- (7) Saturated solution magnesium sulphate.
- (8) Solution of mercuric iodide in acetic acid; formula¹ KI , 33.2 grams Hg Cl_2 , 13.5 grams. Strong $\text{HC}_2\text{H}_3\text{O}_2$, 20.0cc. Water, 64.0cc.

Alcohol, ether, and many solutions of mineral salts, hydrochloric, and other acids were also tried as precipitants for albumen, but none of them presented any advantages which would make a detailed account of the experiments of any interest.

Table No. 9 contains a record of the experiments which led to the adoption of 1cc. acetate of lead solution, or 1cc. acid mercuric nitrate, as the best amount of each for 50cc. of milk.

Nearly all the polarisations were made in a 400mm. tube. From two to four observations were made with each sample. An average of these readings was taken for each determination. In the calculations the value of $(a)_p$ was taken at 53 instead of 52.5, the number which subsequent investigations have led me to believe more exact. The instrument employed was a "Laurent Large Model" polariscope.

In all cases the volume of the solution was corrected for the volume of the precipitated caseine. The volume was assumed to occupy 2cc. for each 50cc. milk.

Since in the Laurent instrument the weight of sucrose in 100cc. to read even degrees on the scale is 16.19 grams [$(a)_p=66.67$], it follows that the weight of lactose in 100cc. to read one degree on the scale for each percent. lactose present would be 16.19: $x=53$: 66.67; $x=20.37$.

If 52.5 be taken as the value of $(a)_p$ for lactose, then $x=20.56$.

In table No. 9, A indicates acetic acid, Pb basic acetate of lead, MR acid mercuric nitrate, &c. The letters C and H indicate the temperature; C denoting the ordinary temperature of the room, and H that the sample was heated to 100° C. and cooled before filtering.

¹Jour. de Pharm. et de Chim., vol. 10, p. 108.

The numbers obtained by extraction with alcohol are taken as the basis of comparison, not because I believe them to be more reliable, but because that method is the one generally employed in the estimation of milk sugar.

In the alcohol extraction the milk was evaporated to dryness in a thin glass capsule, the dish and dried residue pulverized in a mortar, washed with ether into a continuous extraction apparatus, exhausted with ether, and then with 80 per cent. alcohol for ten hours.

Duplicate analyses are indicated in the table by the small brackets.

TABLE No. 9.—Percentage of milk sugar.

Number.	Per cent. lactose extracted by alcohol.	Reagents employed in precipitating albumens.							
		Pb 1cc.	Pb 2cc.	Pb 3cc.	Pb 4cc.	Pb 5cc.	A 5cc.	Other reagents.	
		Per cent. lactose.							
1	4.57			(10 ^{cc.})	3.67	4.23			
2	4.52				2.45	3.57	4.14		
3	4.46	4.48					3.57		
4	3.92	4.19				3.35			
5	4.35		3.55				4.32		
6	3.71	4.01					3.00		
7	4.10	{ 4.63					{ 4.44		
		{ 4.96 H					{ 4.68 H		
8	4.16	{ 4.29					{ 3.80		
		{ 4.33					{ 3.67 H		
9	4.48	{ 4.59					{ 4.04		
		{ 4.58 H					{ 4.12 H		
10	4.10	4.12 H					3.47 H		
11	4.60	4.87 H			(4 ^{cc.})		4.44 H		
12	4.77	5.02 H	4.82 H	4.50 H	4.31 H			H ₂ SO ₄	
							4 ^{cc.}	5 ^{cc.}	6 ^{cc.}
13	4.25	4.25	3.75	3.38	3.38		4.70 H	4.76 H	8 ^{cc.}
							3 ^{cc.}		
14	4.22	4.90 H	4.58 H				3.97	3.89	4.78 H
15	3.14	{ 4.40						HNO ₃	
16	3.30	{ 4.32 H					4.68 H	4.66 H	4.50 H
17	4.72	4.45	4.18	3.87	3.65	3.26			
							3.96	3.98	3.88
18	4.88	{ 4.87					{ 4.37	{ 3.98	
		{ 4.87 H					{ 4.25	{ 3.88 H	
19	4.31	{ 4.71					{ 4.43	4.27	
20	4.39	{ 4.86 H					{ 4.51	4.43 H ₂ SO ₄	
21	4.70	4.11					{ 4.43 H	{ 4.59	
22	4.96	4.17						{ 4.59 H	
		{ 4.93					{ 4.45	4.69	
23	4.60	{ 4.97 H					{ 4.43 H	4.67 H	
		{ 4.41					{ 3.86	3.94	
24	4.74	{ 4.45					{ 3.86	3.90	
		{ 4.41					{ 4.21	4.32	
25	4.59	{ 4.45 H					{ 4.35 H	4.44 H	
		{ 4.33					{ 3.34	4.03	
26	4.39	{ 4.37 H	{ NaCl	1 ^{cc.} MR			{ 3.93 H	4.10 H	
27	4.60	{ 4.29 H	4.29 H					4.10 H	
28	4.26	4.18 H	4.66						
		3.67 H	4.09						

Remarks on Table No. 9.—The results obtained by using various other reagents for the precipitation of the caseine, viz, MgSO₄, CuSO₄, HCl, &c., have not been entered in the table. In none of these cases was there sufficient encouragement to warrant an extended trial. In most cases the precipitation was slow or imperfect, and the filtration difficult.

One important fact should not be overlooked, viz, that any excess of basic plumbic acetate causes a rapid decrease in the rotatory power of the solution; whether this decrease is due to precipitation of the sugar

or solution of the albumens does not clearly appear. Illustrations of this decrease are seen in analyses 2, 12, 13, and 17.

It seems to make little difference whether the precipitation is made hot or cold. The question of temperature is set forth in greater detail in the next table. From all the experiments made it clearly appeared that the best optical results are obtained by the use of a minimum quantity of basic lead acetate, or of either the acid mercuric nitrate or iodide. For 50cc. to 60cc. of milk, 1cc. of the lead acetate or mercuric nitrate solution of the strength noted, and 25cc. of the mercuric iodide solution are the proper quantities. It makes no difference, however, if a large excess of the two latter reagents is employed. Of the three the last is to be preferred.

In Table No. 10 will be found the results of the comparative determinations of milk sugar by extraction with alcohol, by precipitation with 1cc. basic lead acetate, and the same with 1cc. acid mercuric nitrate, hot and cold, to each 60cc. of milk.

In many of the analyses the large differences in results by the three methods show a fault of manipulation, but all the results have been given without selection.

TABLE NO. 10.—*Percentage of milk sugar.*

No.	Reagents employed.					No.	Reagents employed.				
	Extracted by alcohol.	C. Pb 1cc.	H. Pb 1cc.	C. MR1cc.	H. MR1cc.		Extracted by alcohol.	C. Pb 1cc.	H. Pb 1cc.	C. MR1cc.	H. MR1cc.
	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>		<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>
1	4.55	4.74	4.92	34	4.37	4.65	4.93	4.93
2	4.10	4.22	4.50	35	4.52	4.27	4.41	4.56
3	4.51	4.54	4.22	4.68	4.62	36	4.88	4.83	4.93	5.17
4	4.36	4.55	4.53	4.89	4.90	37	4.61	4.30	4.43	4.57
5	4.05	4.14	4.09	4.48	4.39	38	4.79	4.59	4.67	4.91
6	3.84	3.84	3.98	3.98	39	4.67	4.26	4.41	4.51
7	4.52	4.67	4.73	5.01	5.00	40	4.79	4.64	4.74	4.94
8	4.25	4.21	4.26	4.51	41	3.95	4.10	4.26	4.38
9	4.45	4.61	4.54	4.87	4.87	42	4.00	4.61	4.61	4.77
10	4.92	5.20	5.22	5.43	5.47	43	4.63	4.24	4.37	4.57
11	3.84	3.72	4.00	3.96	44	4.77	4.64	4.70	4.94
12	4.53	4.61	4.64	4.87	4.85	45	4.85	4.53	4.73
13	4.57	4.54	4.55	4.91	4.84	46	4.71	4.67	4.93
14	4.66	4.29	4.45	4.63	47	4.34	4.06	4.12	4.40
15	4.17	3.65	3.75	3.95	3.87	48	4.05	4.67	4.77	4.83
16	5.02	4.66	4.64	4.86	4.86	49	3.67	4.12	4.18	4.36
17	4.68	4.03	3.94	4.39	4.37	50	3.78	4.58	4.62	4.82
18	4.23	3.82	3.89	4.08	4.02	51	4.19	4.27	4.57	4.53
19	4.96	4.70	4.84	5.04	5.04	52	3.83	4.68	4.78	4.97
20	4.85	4.39	4.41	4.53	4.65	53	3.86	3.97	4.07	4.21
21	4.63	4.47	4.47	4.69	4.67	54	4.59	4.59	4.61	4.63
22	4.47	4.39	4.45	4.67	4.71	55	4.02	4.26	4.36	4.40
23	4.46	4.23	4.31	4.65	4.63	56	4.36	4.62	4.76	4.94
24	4.47	4.59	4.67	5.01	4.95	57	4.20	4.18	4.28	4.48
25	4.40	4.41	4.55	4.45	58	4.09	4.52	4.56	4.74
26	4.85	4.67	4.73	4.97	59	4.09	4.28	4.46
27	4.45	4.21	4.33	4.57	60	4.12	4.49	4.81
28	4.44	3.98	4.10	4.28	61	4.20	4.33	4.41
29	4.10	4.21	4.55	62	4.45	4.25	4.77
30	4.38	5.57	4.69	4.89	63	4.33	4.09	4.37
31	4.20	4.21	4.37	4.57	64	4.62	4.33	4.99
32	4.69	4.59	4.67	4.89						
33	4.52	4.27	4.41	4.41	Av	4.33	4.34	4.38	4.58	4.63

In the following table will be found the percentage of milk sugar obtained by using varying quantities of the mercuric iodide reagent, and a comparison of the results obtained with those given by the use of acid mercuric nitrate and basic plumbic acetate:

TABLE NO. 11.—*Percentage of milk sugar.*

Number.	Reagents-employed.					
	Pb.	MR.	Mercuric iodide.			
			20cc.	25cc.	30cc.	35cc.
	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>
1	4.28	4.48	4.56	4.56	-----	-----
2	4.46	4.57	4.62	4.66	-----	-----
3	4.37	4.65	4.63	4.63	4.60	4.65
4	4.37	4.53	4.60	4.53	4.63	4.60
5	4.38	4.63	4.50	4.53	4.53	4.59
6	4.33	4.67	4.43	4.53	4.60	4.66
7	4.30	4.67	4.67	4.67	4.59	4.57
8	4.33	4.59	4.50	4.53	4.50	4.59
9	4.27	4.60	4.63	4.60	4.66	4.66
Av.	4.34	4.60	4.57	4.58	4.61	4.62

ALBUMEN REMAINING IN FILTRATE FROM LEAD ACETATE AND MERCURIC IODIDE SOLUTIONS.

From the fact that the polariscopic readings show that solutions of milk prepared with lead acetate have a lower rotating power than those prepared with mercury salts, it is to be inferred that the lead reagent either leaves certain soluble and transparent kinds of albumen in solution, or else dissolves a portion of those which are at first precipitated. To test the accuracy of this supposition a few analyses were made to determine the amount of albumen left in the filtrate from the lead and mercury reagents. At the same time different quantities of the mercuric iodide solution were used, in order to determine the amount which would give the best results. For 60cc. milk the quantity of mercuric iodide to be used should be 25cc. to 30cc.

In the following table will be found the percentages of albumen in the whey after precipitating with the reagents noted and filtering. Ten cubic centimeters of the filtrate were evaporated to dryness in a thin glass dish, and the dried residue (with the glass) burned with soda lime. The calculated nitrogen was then multiplied by 6.25 and the product taken as the percentage of albumen :

TABLE NO. 12.—*Per cent. albumen in filtrate.*

From Pb.	From HgI ₂ . 15cc.	From HgI ₂ . 20cc.	From HgI ₂ . 25cc.	From HgI ₂ . 30cc.	From HgI ₂ . 35cc.
.0865	.1950	.0865	.0865	.0562	.0865
.1130	.0674	.0865	.0865	.0865	.0865
.1130	.0674	.0562	.1130	.0562	.0312
.0865	.0674	.0562	.1130	.0562	.0562
.1130	.0674	.0562	.0312	.0865	.0562
.1130	.0090	.0865	.0300	.0865	.1412
.1130	-----	.1412	.1130	.1412	.0562
.1950	-----	.1412	.1130	.1412	.1412
.1130	-----	.1412	.0865	.1412	.0865
.1412	-----	.1130	.1412	.1130	.0865
.1130	-----	.1362	.0090	-----	-----
-----	-----	.1250	-----	-----	-----
-----	-----	.0090	-----	-----	-----
-----	-----	.0090	-----	-----	-----
Av... 1182	.0789	.0888	.0839	.0964	.0828

In Table No. 13 will be found percentages of albumen remaining in filtrate from lead acetate precipitation of forty-two samples taken from those represented in Table No. 10. From these two tables it is at once seen that the quantity of lævo-rotatory matter remaining in milk after treatment with basic lead acetate is much greater than in those samples treated with the two mercuric salts. This explains at once the higher per cent. of milk sugar obtained by using the last-named reagents, and shows that the use of lead acetate as a clarifying agent must be abandoned:

TABLE NO. 13—*Per cent. albumen after precipitation by lead acetate.*

No.	Per cent.	No.	Per cent.	No.	Per cent.
1	.250	16	.237	31	.329
2	.366	17	.237	32	.305
3	.135	18	.169	33	.305
4	.272	19	.103	34	.237
5	.134	20	.271	35	.305
6	.239	21	.237	36	.339
7	.301	22	.271	37	.237
8	.305	23	.235	38	.374
9	.237	24	.271	39	.203
10	.339	25	.237	40	.373
11	.271	26	.237	41	.305
12	.305	27	.271	42	.339
13	.267	28	.339		
14	.237	29	.350	Av.	.278
15	.271	30	.374		

COMPARISON OF RESULTS OBTAINED BY EXTRACTION WITH ALCOHOL AND POLARIZATION.

By consulting Table No. 10, it will be seen that the percentage of sugar obtained by extraction with alcohol is practically the same as that got by polarization of the lead acetate filtrate.

Thus, the mean percentage of sugar by alcohol (65 analyses) is 4.32; by lead acetate, cold (53 analyses) is 4.34; by lead acetate, hot (64 analyses) is 4.38; by mercuric nitrate, cold (61 analyses) is 4.58; by mercuric nitrate, hot (24 analyses) is 4.63.

If now the milk sugar, as has already been intimated, exists in an anhydrous state after extraction with alcohol, the percentage of it after the addition of the molecule of water would be increased. Thus molecular weight of anhydrous milk sugar, 342: molecular weight of the hydrous 360=4.38: x , whence the value of $x=4.61$. This agrees very nearly with the number obtained by acid mercuric nitrate.

By a study of Table No. 13 it is found that the mercuric iodide gives nearly the same rotatory power as mercuric nitrate, and also by combustion the filtrates from the milks clarified by lead acetate contain more albumen than those prepared with mercuric iodide. There is, therefore, every reason for believing that the numbers given by the mercury salts are nearer the truth than those from the lead.

It may be urged that the increased rotatory power observed by the mercury salts is due to the conversion by the dilute acids of a part of the lactose into galactose, which has a rotatory power greater than that of milk sugar. But when it is remembered that the quantity of acid introduced is extremely minute, that the samples need not be warmed,

that they can be filtered and polarized within a few minutes of the time of the introduction of the reagents, the suggestion is seen to be of no force.

For example, in the acid-mercuric nitrate it was found that the percentage of sugar was the same whether one, five, or ten cubic centimeters of the reagent were employed, and whether it was polarized immediately or after heating and cooling. It is evident that 1cc. of the reagent, containing less than a half cubic centimeter of nitric acid and diluted in 100cc. of liquid, could not exert any notable effect on the rotatory power of the solution.

In the mercuric iodide solution 20cc. of acetic acid are used for every 660cc. of the reagent.

Thirty cubic centimeters of this reagent contain, therefore, about 1cc. of acid. This in 100cc. of liquid, immediately filtered and polarized, could not affect in any marked degree the rotatory power.

Since combustion with soda-lime shows that the filtrate from the mercuric iodide sample is practically free from albumen, it is evident that the numbers obtained in this way must be a near approximation to the truth.

THE PROCESS OF ANALYSIS.

The reagents, apparatus, and manipulation necessary to give the most reliable results in milk sugar estimation are as follows:

Reagents.—(1) *Basic plumbic acetate*, specific gravity 1.97. Boil a saturated solution of sugar of lead with an excess of litharge, and make it of the strength indicated above. One cubic centimeter of this will precipitate the albumens in 50cc. to 60cc. of milk.

(2) *Acid mercuric nitrate*, dissolve mercury in double its weight of nitric acid, specific gravity 1.42. Add to the solution an equal volume of water. One cubic centimeter of this reagent is sufficient for the quantity of milk mentioned above. Larger quantities can be used without affecting the results of polarization.

(3) *Mercuric iodide with acetic acid* (composition already given).

Apparatus.—(1) Pipettes marked at 59.5cc., 60cc., and 60.5cc. (2) Sugar flasks marked at 102.4cc. (3) Filters, observation tubes, and polariscope. (4) Specific gravity spindle and cylinder. (5) Thermometers.

MANIPULATION.

(1) The room and milk should be kept at a constant temperature. It is not important that the temperature should be any given degree. The work can be carried on equally well at 15° C., 20° C., or 25° C. The slight variations in rotatory power within the above limits will not affect the result for analytical purposes. The temperature selected should be the one which is most easily kept constant.

(2) The specific gravity of milk is determined. For general work this is done by a delicate specific gravity spindle. Where greater accuracy is required use specific gravity flask.

(3) If the specific gravity be 1.026 or nearly so, measure out 60.5cc. into the sugar flask. Add 1cc. of mercuric nitrate solution or 30cc. mercuric iodide solution and fill to 102.4cc. mark. The precipitated albumen occupies a volume of about 2.4cc. Hence the milk solution is really 100cc. If the specific gravity is 1.030 use 60cc. of milk. If specific gravity is 1.034 use 59.5cc. of milk.

(4) Fill up to mark in 102.4cc. flask, shake well, filter, and polarize.

NOTES.

In the above method of analysis the specific rotatory power of milk sugar is taken at 52.5, and the weight of it in 100cc. solution to read 100 degrees in the cane sugar scale at 20.56 grams. This is for instruments requiring 16.19 grams sucrose to produce a rotation of 100 sugar degrees. It will be easy to calculate the number for milk sugar whatever instrument is employed.

Since the quality of milk taken is three times 20.56 grams, the polariscopic readings divided by 3 give at once the percentage of milk sugar when a 200mm. tube is used.

If a 400mm. tube is employed, divide reading by 6; if a 500mm. tube is used, divide by 7.5.

Since it requires but little more time, it is advisable to make the analysis in duplicate, and take four readings for each tube. By following this method gross errors of observation are detected and avoided.

By using a flask graduated at 102.4 for 60cc. no correction for volume of precipitated caseine need be made. In no case is it necessary to heat the sample before polarizing.

ESTIMATION OF THE ALBUMINOIDS.

The albuminoids in milk are most easily estimated by combustion with soda-lime or by previous conversion into ammoniac sulphate and subsequent distillation from an alkaline liquid.

(1) *Combustion with soda-lime*—From 4 to 5 grams of milk measured from a weighing flask are evaporated to dryness in a schälchen either alone or with sand, gypsum, pumice-stone or asbestos. When dry the whole is rubbed up in a mortar, transferred to a combustion tube, and burned in the usual way. The nitrogen calculated from the ammonia formed multiplied by 6.25 gives the total albuminoids.

(2) The estimation of the albuminoids by Kjeldahl's method is so well understood that it will not be necessary to describe it here.

Following are the results of the analyses of milks made in this laboratory.

In table No. 14 are the results of the daily analyses of milk from the Maythorpe Dairy.

In table No. 15 are the numbers obtained with milks from various sources.

In these analyses the fat was estimated by the modified Soxhlet method, the sugar by the optical method, and the albuminoids by combustion with soda-lime.

TABLE NO. 14.—*Analyses of milk from Maythorpe Dairy.*

[All these samples were bought from D. M. Nesbit, College Station, Md.]

Number.	Specific gravity at 15° C.	Water.	Fat arcometric.	Albuminoids.	Sugar.	Ash.	Total solids.
		<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>
1.....	1.0348	87.31	2.57	4.60	.73	12.69
2.....	1.0340	86.96	2.81	5.00	.74	13.04
3.....	1.0333	87.62	3.64	2.61	4.80	.73	12.38
4.....	1.0339	4.12	2.84	4.85	.72
5.....	1.0326	87.69	4.00	2.72	5.05	.72
6.....	1.0316	87.08	3.92	2.63	5.14	.72	12.92
7.....	1.0337	87.27	2.80	5.19	.72	12.73
8.....	1.0362	87.98	3.21	2.77	5.10	.74	12.02
9.....	1.0341	86.97	3.66	2.75	5.19	.72	13.03
10.....	1.0333	87.62	3.98	2.78	5.27	.71	12.98
11.....	1.0315	87.89	3.51	2.80	4.99	.66	12.11
12.....	1.0337	87.54	4.25	2.80	4.99	.73	12.46
13.....	1.0334	86.09	4.62	2.98	5.02	.77	13.91
14.....	1.0368	85.44	3.92	4.65	.85	14.56
15.....	1.0328	86.73	3.04	5.00	.72	13.27
16.....	1.0358	84.58	4.73	4.05	4.67	.94	15.42
17.....	1.0335	87.84	4.98	3.15	4.92	.73	12.16
18.....	1.0325	87.33	2.63	5.04	.71	12.67
19.....	1.0323	86.60	2.03	4.95	.71	13.40
20.....	1.0353	86.24	4.13	4.70	1.21	13.76
21.....	1.0328	88.56	4.06	2.98	4.75	.55	11.44
22.....	1.0368	84.03	4.51	4.55	1.15	15.97
23.....	1.0328	84.64	5.22	2.84	4.90	.61	15.36
24.....	1.0325	88.10	4.24	2.73	5.04	.72	11.90
25.....	1.0329	86.17	2.76	4.99	.74	13.83
26.....	1.0349	85.46	4.74	3.64	4.95	.86	14.54
27.....	1.0323	86.59	2.87	4.82	.64	13.41
28.....	1.0369	84.93	3.61	4.38	.81	15.07
29.....	1.0330	85.73	4.92	2.98	5.14	.58	14.27
30.....	1.0344	86.60	2.59	5.17	.68	13.40
31.....	1.0339	86.85	3.18	5.00	.62	13.15
32.....	1.0363	85.67	3.92	4.70	.85	14.33
33.....	1.0324	86.55	2.80	4.84	.70	13.45
34.....	1.0369	83.75	4.73	3.69	.84	16.25
35.....	1.0339	85.66	3.29	5.13	.68	14.34
36.....	1.0346	5.08	3.11	5.02	.64
37.....	1.0350	85.67	4.87	2.94	5.67	.74	14.33
38.....	1.0340	4.75	3.22	5.02	.68
39.....	1.0340	87.15	4.47	2.76	5.30	.70	12.85
40.....	1.0355	88.98	4.78	2.76	5.57	.63	11.02
41.....	1.0325	87.71	4.00	2.69	5.02	.56	12.29
42.....	1.0333	87.04	4.11	3.04	4.75	.64	12.96
43.....	1.0338	85.49	5.25	2.84	4.90	.80	14.51
44.....	1.0318	86.92	4.26	2.87	4.80	.70	13.08
45.....	1.0343	86.10	4.66	2.69	4.89	.70	14.90
46.....	1.0343	87.18	3.75	2.59	5.03	.66	12.82
47.....	1.0328	87.44	3.57	2.66	5.01	.72	12.56
48.....	1.0334	86.79	4.03	2.80	4.80	.65	13.21
49.....	1.0342	85.85	4.81	3.15	4.97	.75	14.15
50.....	1.0327	87.87	4.18	2.80	4.80	.72	12.13
51.....	1.0338	86.56	4.02	2.87	5.19	.61	13.44
52.....	1.0319	86.10	4.96	2.63	5.20	.63	13.90
53.....	1.0319	87.63	3.38	2.76	4.95	.70	12.37
54.....	1.0318	87.50	3.51	2.66	4.49	.81	12.50
55.....	1.0343	85.37	4.72	3.15	4.85	.80	14.63
56.....	1.0323	86.85	3.75	2.87	4.77	.71	13.15
57.....	1.0338	86.14	4.08	2.87	5.07	.73	13.86
58.....	1.0382	86.23	4.50	2.66	5.15	.77	13.77
59.....	1.0317	87.82	3.16	2.56	4.85	.68	12.18
60.....	1.0325	86.51	4.21	2.66	4.85	.69	13.49
61.....	1.0354	84.64	3.43	5.15	.84	15.36
62.....	1.0339	86.89	4.00	2.84	5.02	.78	13.11
63.....	1.0340	86.98	3.94	2.80	5.23	.69	13.02
64.....	1.0340	87.74	3.72	2.59	5.27	.69	12.26
65.....	1.0305	87.96	3.13	2.56	4.87	.66	12.04
66.....	1.0331	87.57	3.88	2.63	4.95	.73	12.43
67.....	1.0305	89.20	3.57	2.76	4.33	.73	10.80
68.....	1.0328	89.01	3.26	2.87	4.99	.72	10.99
69.....	1.0314	86.32	3.02	2.73	4.67	.69	13.68
70.....	1.0320	87.22	4.65	2.39	5.27	.69	12.78
71.....	1.0320	86.89	4.53	2.98	4.97	.76	13.11
72.....	1.0334	86.55	4.11	2.87	5.15	.55	13.45
73.....	1.0317	88.35	3.55	2.73	4.57	.55	11.65
74.....	1.0345	87.12	3.59	2.91	5.10	.64	12.88
75.....	1.0310	88.01	3.66	2.69	4.49	.56	11.99
76.....	1.0340	87.62	3.67	2.45	5.40	.70	12.38

TABLE No. 14.—*Analyses of milk from Maythorpe Dairy—Continued.*

Number.	Specific gravity at 15° C.	Water.	Fat arcometric.	Albuminoids.	Sugar.	Ash.	Total solids.
		<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>
77.....	1.0336	86.16	4.75	2.84	5.13	.79	13.84
78.....	1.0320	86.80	4.36	2.45	5.15	.62	13.20
79.....	1.0320	84.73	4.25	2.56	4.90	.54	14.27
80.....	1.0328	86.34	3.50	2.91	4.89	.66	13.66
81.....	1.0318	88.71	3.71	2.59	4.80	.54	11.29
82.....	1.0334	86.94	3.97	2.31	5.49	.57	13.06
83.....	1.0325	86.01	4.58	2.91	5.13	.74	13.99
84.....	1.0336	86.41	4.23	2.63	5.22	.76	13.59
85.....	1.0311	87.73	3.61	2.73	4.60	.70	12.27
86.....	1.0332	87.09	3.79	2.69	4.99	.64	12.91
87.....	1.0321	86.54	4.11	2.63	4.90	.64	13.46
88.....	1.0322	86.73	4.41	2.24	5.30	.66	13.27
89.....	1.0335	85.75	3.85	3.01	5.25	.71	14.25
90.....	1.0312	88.66	4.64	2.34	5.23	.68	11.34
91.....	1.0328	87.87	3.61	2.48	5.25	.68	12.13
92.....	1.0341	86.94	4.06	2.38	5.49	.67	13.06
93.....	1.0322	87.63	3.75	2.56	5.02	.69	12.37
94.....	1.0322	86.59	3.78	2.28	5.29	.58	13.41
95.....	1.0312	88.30	2.73	2.45	4.93	.65	11.70
96.....	1.0322	87.42	4.30	2.45	5.25	.66	12.58
97.....	1.0332	87.65	3.68	2.41	5.27	.69	12.35
98.....	1.0332	87.22	4.35	2.66	5.37	.66	12.78
99.....	1.0313	88.93	4.79	2.59	4.95	.70	12.07
100.....	1.0328	87.29	4.07	2.38	5.37	.72	12.71
101.....	1.0325	88.31	3.05	2.45	5.50	.63	11.69
102.....	1.0345	87.40	3.65	2.41	5.45	.70	12.60
103.....	1.0345	87.78	3.60	2.38	5.33	.72	12.22
104.....	1.0355	88.38	3.97	2.63	5.75	.59	11.62
105.....	1.0330	88.24	3.14	2.63	5.22	.72	11.76
106.....	1.0345	88.60	3.73	2.45	5.67	.72	11.40
107.....	1.0335	88.58	3.06	2.63	5.30	.55	11.42
108.....	1.0317	88.86	3.57	2.24	5.05	.65	11.14
109.....	1.0325	87.89	2.61	2.45	5.07	.60	12.11
110.....	1.0320	88.47	4.02	2.34	5.23	.66	11.43
111.....	1.0320	88.97	4.68	2.52	5.27	.70	12.03
112.....	1.0315	87.85	4.67	2.34	5.07	.68	13.15
113.....	1.0315	87.29	4.19	2.45	4.93	.66	12.71
114.....	1.0367	86.10	4.74	2.84	5.49	.69	13.90
115.....	1.0356	86.61	4.28	3.08	5.35	.76	13.39
116.....	1.0354	87.59	3.13	2.63	5.52	.73	12.41
117.....	1.0336	86.37	4.41	2.52	5.23	.73	13.63
118.....	1.0340	86.17	4.45	2.73	5.25	.74	13.83
119.....	1.0326	86.09	5.12	2.80	5.29	.69	13.91
120.....	1.0338	85.88	5.09	2.69	5.43	.72	14.12
121.....	1.0352	86.91	4.16	2.94	5.30	.74	13.09
122.....	1.0342	87.17	3.80	2.69	5.33	.70	12.83
123.....	1.0352	85.99	4.57	2.80	5.42	.71	14.01
124.....	1.0338	86.64	4.47	2.31	5.24	.72	13.36
125.....	1.0348	87.55	3.78	2.76	5.50	.79	12.45
126.....	1.0343	85.41	4.91	3.01	5.47	.71	14.59
127.....	1.0352	86.47	3.99	2.91	5.32	.72	13.43
128.....	1.0357	87.81	3.15	2.76	5.62	.77	12.19
129.....	1.0336	87.66	4.74	2.91	5.20	.72	12.34
130.....	1.0336	86.32	4.50	2.66	5.27	.71	13.68
131.....	1.0341	85.88	5.02	2.69	5.49	.69	14.12
132.....	1.0351	86.57	4.65	2.69	5.55	.75	13.43
133.....	1.0354	85.81	4.51	2.98	5.58	.76	14.19
134.....	1.0353	87.33	3.66	2.45	5.67	.89	12.67
135.....	1.0346	86.34	4.68	2.63	5.39	.75	13.66
137.....	1.0338	85.48	6.05	2.80	5.39	.79	14.52
138.....	1.0345	86.60	3.74	2.8777	13.40
139.....	1.0315	85.60	5.63	2.4871	14.40
140.....	1.0320	86.85	4.09	2.6373	13.15
141.....	1.0330	87.18	3.49	2.9473	12.82
142.....	1.0340	86.18	4.30	2.9874	13.82
143.....	1.0330	87.54	3.51	3.0460	12.46
144.....	1.0341	86.98	3.82	2.4567	13.02
145.....	1.0320	87.66	6.03	2.3474	12.34
146.....	1.0333	86.65	3.33	2.8766	13.35
147.....	1.0335	86.24	3.57	2.9158	13.76
148.....	1.0350	88.02	2.65	3.0873	11.98
149.....	1.0332	89.38	3.21	2.8759
150.....	1.0344	86.72	3.54	3.1578	13.28
151.....	1.0333	85.34	5.35	2.4570	14.66
152.....	1.0333	86.41	4.18	2.8060	13.59
153.....	1.0342	87.35	3.50	3.0475	12.65
154.....	1.0338	86.66	3.83	2.6664	13.34
155.....	1.0345	87.68	2.91	2.7659	12.32

TABLE No. 14.—*Analyses of milk from Maythorpe Dairy—Continued.*

Number.	Specific gravity at 15° C.	Water.	Fat areometric.	Albuminoids.	Sugar.	Ash.	Total solids.
		<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>
156.....	1.0353	86.43	3.87	2.3770	13.57
157.....	1.0320	86.46	5.29	2.5254	13.54
158.....	1.0335	86.99	3.91	2.6353	13.01
159.....	1.0340	86.89	3.73	3.1958	13.11
160.....	1.0339	86.73	3.97	2.9170	13.27
161.....	1.0333	87.59	3.30	2.6671	12.41
162.....	87.03	3.61	3.1560	12.97
163.....	87.73	3.43	2.6660	12.27
164.....	87.09	3.76	2.9164	12.91
165.....	4.04	2.73
166.....	5.41	2.27
167.....	3.76	3.11
168.....	3.85
169.....	3.94	3.92
Means ...	1.0334	86.95	4.08	2.78	5.05	.70	13.09

TABLE 15.—*Analyses of milk from various sources.*

1*.....	1.0315	88.14	4.73	2.88	4.67	.69	11.86
2*.....	1.0320	5.86	2.84	5.00	.71
3*.....	1.0316	87.48	5.05	2.84	4.75	.73	12.52
4†.....	1.0265	90.91	2.35	2.10	3.77	.62	9.09
5†.....	1.0254	91.50	2.01	1.96	3.63	.52	8.41
6†.....	1.0245	88.76	4.51	2.06	2.02	.56	11.24
7†.....	1.0285	89.84	2.08	2.17	4.23	.69	10.16
8†.....	1.0335	86.97	4.07	2.91	4.92	.68	13.03
Means ...	1.0292	89.09	3.91	2.47	4.12	.65	9.54

* From C. J. Loomis, 1413 Stoughton Hill. † Department lunch room.

‡ Thompson's Dairy.

KOUMISS.

The use of koumiss, both as a beverage and in the sick-room, is rapidly increasing in this country, and for this reason I have thought it would be of interest to add here the results of the investigations on some home-made koumiss.¹

Fermented mare's milk has long been a favorite beverage in the East, where it is known as "koumiss." Although the Tartars and other Asiatic tribes use mare's milk for the manufacture of koumiss, yet it is not the only kind that can be employed. Since the consumption of milk-wine has extended westward cow's milk is chiefly employed for making it both in Europe and America. Mare's milk is considered most suitable for fermentation because of the large percentage of milk-sugar which it contains.

König² gives as the average percentage of milk-sugar in mare's milk 5.31. The same author³ gives as a mean of 377 analyses of cow's milk 4.81 per cent. of lactose. Dr. Stahlberg,⁴ who brought forty mares from the steppes of Russia to Vienna for the purpose of using their milk for

¹ Am. Chem. Jour., vol. 8, p. 200.³ *Op. cit.*, p. 40.² Nahrungsmittel, p. 46.⁴ Tymowskis' Bedeutung des kumys, p. 12.

koumiss, found its percentage of lactose to be 7.26. On the other hand, ordinary mares that were kept at work gave a milk containing only 5.95 per cent. sugar. The quantity of milk-sugar in mare's milk is great, but there is a deficiency of fat and other solids. It appears to contain fully 89 per cent. water, while cow's milk does not have more than 87 per cent.

The process of manufacture is not uniform. In the East the mare's milk is placed in leathern vessels; to it is added a portion of a previous brewing, and also a little yeast. In thirty to forty-eight hours the process is complete. During this time the vessels are frequently shaken.

In the samples analyzed by me the milk was treated with a lactic ferment and yeast. After twenty-four to forty-eight hours' fermentation the koumiss was bottled. The bottles were kept in a cool place, not above 50° F., and in a horizontal position. When shipped to me they were packed in ice. After they were received in the laboratory they were kept on ice until analyzed.

METHOD OF ANALYSIS.

Carbonic dioxide.—The estimation of the carbonic dioxide was a problem of considerable difficulty. It was evidently impracticable to attempt opening the bottle and determining the gas in a portion of the contents. Fortunately I had access to a large balance which would turn with a milligram. On this I weighed the whole bottle, into the cork of which I had inserted a stop-cock such as is sometimes used with a champagne bottle. With the bottle of koumiss were also weighed two drying flasks containing concentrated sulphuric acid with their connections.

Having obtained the weight of the whole, the gas was allowed to escape slowly from the stop-cock and to bubble through the sulphuric acid in the washing flasks.

These flasks, previously to being weighed, were filled with the gas from an ordinary carbonic dioxide generator. After the gas had almost ceased to flow the bottle of koumiss was frequently shaken. It was also placed in a pail of water having a temperature of 30° C. After half an hour the gas ceased to come over.

The whole apparatus was again weighed. The loss of weight gave the quantity of free carbonic dioxide in the sample. After the analysis was completed the volume of the bottle was measured. It is fair to assume that at 30° C. the koumiss still contained an equal volume of dissolved CO₂. In determining the total CO₂ this volume, or its equivalent weight, was added to that obtained by direct determination.

By this method the CO₂ dissolved under pressure in the bottles is estimated separately from that which the koumiss contains in solution under the weight of one atmosphere. Since it is of no importance to separate the gas into these two portions, I have given it altogether in the tables, in volume, by weight; and in percentage by weight.

Acidity.—The samples examined showed under the microscope the acetic ferment, and a portion of the acidity was therefore due to acetic acid. It is the custom in giving the results of analyses of koumiss to represent the whole of the acidity as due to lactic acid. If ordinary yeast is used, and it generally is, it is possible that acetic acid may be formed. This appeared to be the case with the samples in question, since in distilling them a larger percentage of acid was found in the distillate than could have been expected had lactic acid only been present.

I made no attempt to separate these two acids, but estimated the total acidity, and then represented it in terms of both acids.

The direct titration of the lactic acid in the koumiss was attended with such difficulty that the attempt was abandoned. Whatever indicator was employed, the change in color was so obscured that no sharp reaction could be obtained.

To obviate this trouble the koumiss was mixed with an equal volume of saturated solution of magnesium sulphate. After shaking the mixture it was poured through a linen filter. The first portions running through were turbid. After refiltering these the filtrate was quite clear.

Better results were obtained by using with the koumiss equal volumes of alcohol. The filtrate from this mixture was uniformly bright. In this filtrate the acid was estimated by titration with standard sodic-hydrate solution, making the proper corrections for dilution and using phenol-phthalein as an indicator. I would recommend this alcoholic method of clarification to all who may have occasion to determine acid in milk.

Alcohol.—The alcohol was estimated by distilling 500cc. koumiss with 100cc. water until the distillate amounted to 500cc. This, being still turbid, was redistilled with a small quantity of water. The final distillate of 500cc. was used for the estimation of the alcohol in the usual way, viz, by taking its specific gravity and calculating the alcohol from tables.

Milk-sugar.—The milk-sugar was estimated by the method I recommended in a paper read at the Philadelphia meeting of the A. A. A. S.¹

Fat.—Twenty grams of the koumiss were evaporated to dryness in a schälchen, the whole rubbed to a fine powder, and extracted with ether in a continuous extractor. The process of extraction lasted six hours.

Albuminoids.—The albuminoids were estimated by evaporating 5 grams of the material in a schälchen, rubbing to a fine powder with soda-lime, and burning with the same in the usual way.

¹ Am. Chem. Jour., vol. 6, p. 289 *et seq.*

Water.—In a flat platinum dish partly filled with washed and dried sand 2 grams of material were weighed and dried to a constant weight at 100° C. Following are the results of the analyses:

TABLE No. 16.—*Analyses of koumiss.*

No. of analysis.	Weight of koumiss.	Volume of CO ₂ .	Weight of CO ₂ .	CO ₂ by weight.	Acidity as acetic acid.	Acidity as lactic acid.	Alcohol.	Nitrogen.	Albuminoids.	Fat.	Water.	Milk sugar.
	Grams.	Litres.	Grams.	Pr. ct.	Pr. ct.	Pr. ct.	Pr. ct.	Pr. ct.	Pr. ct.	Pr. ct.	Pr. ct.	Pr. ct.
1.....	747.415	2.543	5.009	.67	.31	.47	.87	.431	2.69	2.21	88.81	4.33
2.....	729.376	3.140	6.186	.85	.31	.47	.66	.412	2.58	2.15	89.53	4.31
3.....	768.575	3.179	6.269	.82	.34	.51	.69	.483	3.02	2.07	89.15	4.33
4.....	736.035	3.281	6.463	.88	.30	.45	.81	.482	3.01	1.99	89.31	4.43
5.....	746.187	3.579	6.850	.91	.32	.48	.86	.423	2.64	1.67	89.97	4.43
6.....	750.247	2.973	5.757	.77	.27	.43	.70	.450	2.81	1.75	89.87	4.33
7.....	738.840	3.204	6.313	.85	.33	.49	.73	.462	2.89	2.44	89.01	4.48
8.....	752.550	3.263	6.428	.8577	.450	2.81	2.34	88.87
Mean83	.31	.47	.76	.449	2.56	2.08	89.32	4.38

It will be of interest to compare these results with those obtained by other analysts, both with koumiss from mare's milk and from other sources. As a mean of fourteen analyses of mare's milk koumiss, König¹ gives the following figures, viz:

	Per cent.
Alcohol.....	1.84
Lactic acid.....	0.91
Milk sugar.....	1.24
Albuminoids.....	1.97
Fat.....	1.26
Ash.....	0.30
Carbonic dioxide.....	0.952

The mean of two samples of koumiss made of cow's milk is given by the same author, as follows:

	Per cent.
Alcohol.....	2.64
Lactic acid.....	0.80
Milk sugar.....	3.10
Albuminoids.....	2.02
Ash.....	0.45
Carbonic dioxide.....	1.03

In nine analyses of koumiss² probably made of cow's milk the means are as follows:

	Per cent.
Alcohol.....	1.38
Lactic acid.....	0.82
Milk-sugar.....	3.95
Albuminoids.....	2.89
Fat.....	0.88
Ash.....	0.53
Carbonic dioxide.....	0.77

¹ König, *Nahrungsmittel*, vol. 1, p. 68.² *Op. cit.*, vol. 1, p. 68.

Interesting analyses of koumiss prepared from mare's milk have also been made by Dr. P. Vieth.¹

The mares from which the milk was taken were on exhibition at the London International Exposition for 1884. These animals were obtained from the steppes of Southeastern Russia. The mares were from five to six years old, and were cared for and milked by natives of the country from which they were taken. When milked five times daily the best of these mares gave from four to five litres of milk. It is to be regretted that the milk-sugar, the most important ingredient of milk in respect of koumiss manufacture, was estimated by difference. Eleven analyses of the mixed milk gave the following numbers:

Table of analyses.

	Specific gravity.	Water.	Fat.	Albuminoids.	Milk sugar.	Ash.
		<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Minimum.....	1.0335	89.74	0.87	1.71	6.30	0.26
Maximum.....	1.0360	90.41	1.25	2.11	6.82	0.36
Mean.....	1.0349	90.06	1.09	1.89	6.65	0.31

The koumiss from the above milk had the following composition:

Sample No.	Water.	Alcohol.	Fat.	Albumi- noids.	Lactic acid.	Milk- sugar.	Ash.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
1.....	90.90	2.47	1.08	2.25	0.64	2.21	0.36
2.....	91.95	2.70	1.13	2.00	1.16	0.69	0.37
3.....	91.79	2.84	1.27	1.97	1.26	0.51	0.36
4.....	91.87	3.29	1.17	1.99	0.96	0.39	0.33
5.....	92.38	3.26	1.14	1.76	1.03	0.09	0.34
6.....	92.42	3.29	1.20	1.87	1.00	0.00	0.35
7.....	91.42	2.25	1.22	1.75	0.70	2.30	0.36
8.....	92.04	2.84	1.10	1.89	1.06	0.73	0.34
9.....	91.99	2.81	1.44	1.69	1.54	0.19	0.34
Mean.....	91.87	2.86	1.19	1.91	1.04	0.79	0.35

Collecting the above means together, we have the following comparative table:

Sample No.	Alcohol.	Lactic acid.	Sugar.	Albumi- noids.	Fat.	CO ₂ .	Water.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
1.....	1.84	0.91	1.24	1.97	1.26	0.95	² 92.47
2.....	2.64	0.80	3.10	2.02	0.85	1.03	88.72
3.....	1.38	0.82	3.95	2.89	0.88	0.77	² 89.55
4.....	2.86	1.04	0.79	1.91	1.19	91.87
5.....	0.76	0.47	4.38	2.56	2.08	0.83	89.32

NOTES.—No. 1, mean of 14 analyses of koumiss from mare's milk; No. 2, mean of 2 analyses of koumiss from cow's milk; No. 3, mean of 9 analyses of koumiss, origin unknown, probably from skimmed cow's milk; No. 4, mean of 9 analyses of koumiss made from mare's milk, London Exposition of 1884; No. 5, mean of 8 analyses of koumiss from cow's milk, made by Division of Chemistry, United States Department of Agriculture.

¹ Landw. Versuchs-Stationen, vol. 31, pp. 353 *et seq.*

² By difference.

The comparison of the above results shows that the American koumiss differs from that of other countries in the following points, viz :

(a) The percentage of alcohol is quite low and as a consequence the percentage of sugar is high.

(b) American koumiss contains more fat; showing that it has been made from milk from which the cream had not been so carefully removed as in those milks from which the European koumiss was made. Mare's milk, as will be seen by the above analyses, contains much less fat and more sugar than that of the cow, thus making it more suitable for the production of koumiss. Good cow's milk, however, is suitable for the manufacture of koumiss after most of the cream has been removed. Should it be desired to make a koumiss richer in alcohol, some milk-sugar could be added.

The samples analyzed were kindly furnished me by Mr. Julius Haag, of Indianapolis. This koumiss makes a delightfully refreshing drink. When drawn from the bottle and poured a few times from glass to glass it becomes thick like whipped cream, and is then most palatable. It is much relished as a beverage, and is highly recommended by physicians in cases of imperfect nutrition. Those desiring to study the therapeutic action of koumiss should consult the monographs of Biel,¹ Stahlberg,² Landowski,³ and Tymowski.⁴

CHEESE.

No studies of cheese have been made in this laboratory.

Caldwell⁵ has given a résumé of the subject up to 1882, as follows:

Literature.—The subject of the adulteration of cheese receives only brief mention either in the journals or in monograph works on adulteration of food.

The Analyst⁶ quotes from the Chicago Journal of Commerce the statement that soapstone, soda, and potash are added to cheese.

Hassell⁷ states that cheese is adulterated with potatoes in Thuringia and in Saxony, and that bean meal is sometimes added in the place of potatoes; that Venetian red has been detected in several cases in the coloring of the rind, and as this color sometimes contains lead, and the rind is sometimes eaten, the fraud may be dangerous. He also says it is stated that blue vitriol and arsenic (green?) are sometimes added, perhaps to give the appearance of age to the cheese, but he has never found them.

Ellsner⁸ says that adulterations of cheese are not known. He mentions oleomargarine cheese as an article recently introduced in Germany. Griessmayer⁹ also says that cheese is not adulterated; but he mentions in appropriate terms a practice of soaking certain kinds of cheese, such as Limburger, in urine in order to give

¹ Untersuchungen über den Kumys und den Stoffwechsel während der Kumyscur.

² Kumys, seine physiologische und therapeutische Wirkung. St. Petersburg.

³ Du kumys et de son rôle thérapeutique.

⁴ Zur physiologischen und therapeutischen Bedeutung des Kumys. München.

⁵ Second Ann. Rept. N. Y. S. Bd. of Health, p. 529.

⁶ 1881, p. 29.

⁷ Food and its adulterations, 1876.

⁸ Die Praxis der Nahrungsmittel-Chemiker, 1880.

⁹ Die Verfälschung der wichtigsten Nahrungs- und Genussmittel, 1880.

them in a short time the appearance of ripeness; such cheese can be made to show the reaction for murexide. He mentions the possible occurrence of poisonous metals, as copper, lead, or zinc in cheese, owing to carelessness in keeping it in metallic vessels or wrappings.

Fleischmann¹ quotes the results of Vogel's examination of cheese for lead; beyond 2 inches from the rind no lead was found, even in cases of cheese wrapped in very inferior tinfoil containing much lead; but in such cases lead was detected in the portions of the cheese immediately under the rind; 0.56 per cent. of lead was found in one instance in the rind of a cheese wrapped in tinfoil containing 15 per cent. of lead. Such cheeses are so little used in this country, however, that this matter has no general importance; but the information may serve as a warning to those who do eat them to be careful of eating the rind.

The same author mentions also the use of veratrin, sulphate of zinc, and arsenic to give to green cheese the strong biting flavor of old cheese, and the addition of blue vitriol to the milk in order to prevent huffing of the cheese.

Liebermann² mentions the danger in metallic wrappings, and states that verdigris is sometimes sprinkled over the cheese to give it the appearance of age. Blythe³ states that washes containing arsenic and lead have often been applied to ward off flies, and as some people eat the rind, such practices may be dangerous.

Lard cheese.—About ten years ago dairymen were much concerned lest the manufacture of cheese from skimmed milk and oleomargarine should seriously injure the reputation of American cheese abroad, and in that way hurt the dairyman's business here. Whatever chance this mode of making cheese may have had for success, it is now quite supplanted by the lard cheese which is made at over twenty factories in this State, under patents issued to H. O. Freeman in 1873 and to William Cooley in 1881.

In this process an emulsion of lard is made by bringing together in a "disintegrator" lard and skimmed milk, both previously heated to 140° Fahr. in steam-jacketed tanks; the "disintegrator" consists of a cylinder revolving within a cylindrical shell: the surface of the cylinder is covered with fine serrated projections, each one of which is a tooth with a sharp point; as this cylinder revolves, rapidly within its shell, the mixture of melted lard and hot skimmed milk is forced up in the narrow interspace; and the lard becomes very finely divided and most intimately mixed or "emulsionized" with the milk. This emulsion consists of from two to three parts of milk to one of lard; it can be made at one factory and taken to another to be used for cheese, but it is usually run at once into the cheese vat.

In making the cheese a quantity of this emulsion containing about 80 pounds of lard is added to 6,000 pounds of skimmed milk and about 600 pounds of buttermilk in the cheese vat, and the lard that does not remain incorporated with the milk or curd, usually about 10 pounds, is carefully skimmed off. These quantities of the materials yield 500 to 600 pounds of cheese, containing about 70 pounds of lard, or about 14 per cent.; about half of the fat removed in the skimming of the milk is replaced by lard (Munsell). It is claimed that no alkali or antiseptic is used, and that only the best kettle-rendered lard can be employed, because of the injurious effect of any inferior article on the quality of the cheese, and that before even this lard is used it is deodorized by blowing steam, under 80 pounds' pressure, through it for an hour.

According to many witnesses the imitation is excellent, for experts have been unable to pick out lard cheeses from a lot of these and full-cream cheeses of good quality together; and it may therefore be safely presumed that the general public would be quite unable to distinguish one from the other.

¹ Das Molkereiwesen, 1879.

² Anleitung zur Chemischen Untersuchung auf dem Gebiete der Medicinpolizei, Hygiene und forensischen Praxis, 1877.

³ Food.

The statistics of the manufacture of this kind of cheese as gathered from various sources, and partly by Inspector Munsell, are about as follows: Thirteen of the "disintegrators" are in operation, all in this State and none elsewhere. The production of cheese at the twenty-three factories engaged in the manufacture in this State during the six months ending November 1, 1881, was stated to amount to 800,000 pounds. None is made in other States, although it was stated before the Assembly Committee on Public Health,¹ in 1881, that it was made at the West. Before the same committee it was stated that some of the cheese was sold in New York City for consumption, but according to the best of my information, most if not all of it is exported. It is claimed that it brings from eight to ten cents a pound when full-cream cheeses sell at twelve cents, and "full-skim" cheeses at four or five cents; but New York dealers tell the inspector that the cheese brings but four cents a pound when its true character is known, and that it is for exportation only. The inspectors have not been able to find any cheese in the city markets which they had any reason to suppose to be lard cheese.

In two respects this kind of cheese can be considered as a fraud under the new food and drug law, unless sold under its distinctive name. It contains less fat, and fat of a cheaper kind, than the ordinary full-cream cheese contains, and, secondly, there are some grounds for the belief that the fat which is substituted for the butter fat is less wholesome than that. Rubner² in some investigations on the assimilation of various articles of food by the human subject found that lard was less digestible than butter; and the objection to oleomargarine butter on the ground of its indigestibility as compared with genuine butter may apply perhaps with more force to lard cheese; it only remains to determine by experiment whether the digestibility of the substances is increased by the operation of emulsionizing.

Skim cheese ("anti-huff cheese") is made, as is well known, from "full-skimmed" milk, without any attempt to replace the fat removed for butter. It is doubtful whether such cheeses are anywhere sold in a way to deceive consumers as to their character. To prove the quality of these cheeses, and especially to prevent them from puffing out, or "huffing," as it is technically called, from the abnormal generation of gases in the interior before they become fully ripe, patented "anti-mottling" and "anti-huffing" extracts are employed, consisting, it is claimed, only of caustic and carbonated alkali, saltpeter, and a little annat to, for coloring, dissolved in water. A qualitative analysis of one of these extracts by both Mr. Munsell and myself confirm this claim in one case; but another extract, said to be used at the West, was found to consist almost entirely of borax, which is a well-known antiseptic. The quantity of alkali and saltpeter said to be added to the cheese in this operation is small, in all less than five ounces to the milk and sour buttermilk for 100 pounds of cheese, and a portion of this must remain in solution in the whey; and there is no satisfactory evidence that such a quantity of borax as could be added to the cheese without affecting its taste would be prejudicial to the health for any ordinary quantity of cheese eaten. Gruber³ shows that when this substance is taken into the system it seems to leave the organism very quickly and without affecting the system in any injurious manner.

As to the statistics of the manufacture of "anti-huff cheese," it is stated that in the most important section of this State for dairy products 4,500 cheeses of the best quality were made this year of skimmed milk and sour buttermilk with the aid of this extract. Before the assembly committee⁴ it was affirmed that this cheese is consumed to

¹ Fenner Committee. Testimony taken before Assembly Committee on Public Health in the matter of investigation into the subject of the manufacture and sale of oleomargarine-butter and lard-cheese. Hon. M. M. Fenner, chairman, 1881.

² Zeits. für Biologie, vol. 15, p. 115. Bied. Centralblatt, 1881; p. 394.

³ Ber. Chem. Gesel., vol. 14, p. 2290. Zeits. für Biologie, vol. 16, p. 195.

⁴ Loc. cit.

some extent in this country, but most of it is exported. It is claimed that nearly the same prices are obtained for the cheese as for full cream cheese, and that it is a good and wholesome article of food, containing nothing but what is found in other food. On the other hand, it is asserted that the excessive quantity of alkali supposed to be in the cheese makes it unwholesome, and that, like the lard cheese, it is a fraud on the public unless sold under a distinctive name; bringing nearly the prices of a full cream cheese, it is taken by consumers to be such. As to the first point there is no evidence *pro* or *con*, and the presumption is, as above stated, that there is no excessive quantity of alkali in the cheese. As to the latter point, it can be left to the interpretation of the law. Without question, a valuable constituent of the cheese has been removed and nothing of the same character has been substituted for it.

Water and fat determined were in small samples, each one of skim cheese made without anti-huffing extract, and with it. Both samples were taken with an ordinary cheese-tryer by Mr. Freeman, the patentee of the process, and sent through Mr. Munsell to me. The results of this partial analysis are given below:

	Water.	Fat.
	<i>Pr. ct.</i>	<i>Pr. ct.</i>
Anti-huffing cheese.....	47.56	14.48
Ordinary skim cheese.....	47.00	16.77

There is nothing unusual in the composition of these samples as compared with skim cheese in general.

Poisonous cheese.—A sample of cheese, said to have produced sickness on the part of those who ate it, was sent to me by the Secretary of the Board, who received it from Inspector Smith. Cases of so-called “poisonous cheese” occasionally appear in different parts of this country and in other countries. Husemann,¹ quoted by Fleischmann,² mentions a number of instances from all parts of Germany, and also in England and Russia, resulting from eating old and especially sour milk and soft rennet cheeses. Scarcely ever is the result fatal, and recovery is rapid, because the vomiting, which is among the first symptoms, relieves the system of the dangerous matter. Voelcker³ after noticing cases of sickness produced by cheese containing copper or zinc sulphate that had been added often surreptitiously by the dairymaid to prevent “heaving” of the cheese, gives an account of a case where all the cheese of a certain “make” sold in different places produced sickness. The cheese presented nothing abnormal in appearance, but his assistants, on eating less than a quarter of an ounce of it, were taken with violent vomiting and pain in the bowels, and a disagreeable mercurial after-taste was left in the mouth. No metallic poisons could be found in it, however, nor anything else abnormal except an apparently larger quantity than usual of fatty acids, giving a strong acid reaction to the cheese. He suggests that the poison is identical with the so-called sausage poison of German sausages made largely from coagulated blood, and says that a similar poison appears to be generated sometimes in pickled salmon, smoked sprats, pork or tainted meat, and that rancid butter may act as a poison. It disappears from the cheese when quite decayed. I have on previous occasions examined such cheese both chemically and with the microscope, without finding any cause for the physiological effect produced by it, or anything unusual. In one case the cheese was excellent in quality otherwise, but it nevertheless, when eaten to test the truth of the allegation against it, made me quite ill with the usual symptoms for a short time. In the present instance some of the cheese was offered to some kittens which are kept in stock for the use of the anatomical department of the University. At first only one would eat it and that one appeared to be quite sick the

¹ Handb. d. Toxicologie, 1862.

² *Loc. cit.*

³ Journ. Roy. Agric. Soc., vol. 23, p. 346.

next day. After her recovery the same kitten ate of the cheese again without any noticeable ill effects, and several others also ate of it without harm. It appears, therefore, that the illness of the first animal may as well have been caused by over-eating of rich food as by any supposed poisonous character belonging to the cheese.

I examined the cheese for poisonous metals in the rind, and for matters of the nature of alkaloids in the other part; no metals were found. With respect to the second test, a very small quantity of a substance, precipitated by alkali, soluble in ether, and giving with platinum chloride a yellowish flocculent precipitate, was obtained. These reactions indicate an alkaloid, but I have not been able as yet to carry the examination any further, and, moreover, it is not at all unlikely that a substance of the same character may be found in any ripened cheese as one of the normal products of the putrefaction. Therefore, this result obtained with the poisonous cheese can have no significance till normal cheese has been examined in the same manner without finding any evidence of the presence of alkaloids. For the present, therefore, we can only repeat what others have said who have given this matter their attention, that the cause of this peculiar property of cheese is probably an unknown organic substance, resulting from an abnormal process of ripening.

Fickert¹ gives the results of some recent legal investigations on this subject. Cheese is so seldom the object of adulteration that when lately the daily papers stated that it was sometimes treated with urine in order to give it more quickly the desired odor and taste, it was considered as an isolated case. More worthy of note, therefore, was the discovery in a trial at Frankenberg, in Saxony, that mashed boiled potatoes had been used as an adulterant of cheese. This adulteration had already been discussed by Popperheim, and that, too, in cheese made especially for home consumption and not intended for commerce. This adulterant is easily detected by the microscope and by iodine.

Since the intrinsic value of cheese depends largely on its high content of albuminoids, viz, about 30 per cent., and since potatoes contain not much over a per cent. of these bodies, it is easily seen how greatly the value of the cheese is impaired by such an admixture.

TYROTOXICON.

The poisonous substance which sometimes is developed in cheese and milk has been isolated by Vaughn.² This substance, the chemical nature of which is not yet fully understood, has been found in cheese, milk, ice-cream, and oysters. For an account of its toxic properties consult Dr. Vaughn's papers.

¹Chem. Centralblatt, 1886, p. 956; Rep. d. ver. anal. Chem., vol. 6, p. 486.

²Paper read at Buffalo meeting A. A. A. S., Aug., 1886, Chem. News, 1886; Medical News, April 2, 1887, p. 369.

ADDENDA.

ESTIMATION OF FAT IN MILK.

Morse and Piggot¹ describe a method of estimating fat in milk by previous desiccation with dehydrated sulphate of copper. About 20 grams of the dried copper sulphate are placed in a porcelain mortar and 10^{cc} of milk added to it, being careful that none of the milk comes in contact with the mortar. The milk is dried in a very few moments, and the mass is then rubbed up with a little clean sand and transferred to an extraction tube. The mortar is then washed two or three times with from 10 to 15^{cc} of benzine, and the fat is extracted by treating in the extraction tube twelve times with the same quantity of benzine. The flask which has received the solution of butter is now placed on a water-bath and the volume of the solution reduced to 10^{cc} or less. The butter fat is now saponified with 20^{cc} of half normal solution of caustic potash. The excess of alkali is determined by a standard solution of hydrochloric acid. The results obtained by this method agree closely with the gravimetric determinations.

ESTIMATION OF WATER IN MILK.

The determination of water in milk is made in the following manner, described by F. G. Short²: About 2 grams of milk are placed in a Hofmeister capsule (schälchen) and dried at 110° C. in an oven containing a solution of chloride of calcium, boiling at 110° C.

When the fat is to be subsequently estimated the capsule is wrapped in a piece of prepared cheese-cloth, crushed between the fingers, and placed in an extraction tube.

SUBSTANCES SOMETIMES ADDED TO MILK TO MASK THE REMOVAL OF THE CREAM AND ADDITION OF WATER.

Where much water is added sugar is most frequently used to increase the specific gravity to the normal number.

Chalk, salt, annatto, turmeric, gum, dextrine, and cerebral matter have also been found in milk by Professor Weber.

¹Amer. Chem. Journ., vol. 9, p. 108.

²Amer. Chem. Journ., vol. 9, p. 100.

MILK ADULTERANT.

Felix Lengfeld, of San Francisco, has communicated to me the composition of a milk adulterant which has been largely used in San Francisco. The mixture consists of common salt, saltpeter, saleratus, a trace of caustic soda, and a large quantity of sugar. The color is imparted by caramel. These bodies are dissolved in an excess of water and the solution used for adulterating milk in any desired quantity.

OCCURRENCE OF ULTRAMARINE AS AN ADULTERANT IN MILK.

Thoms¹ has analyzed a milk which had a chalky appearance, and on standing showed an accumulation of a bluish liquid at the surface. Ultra-marine was found present in the proportion of 82.3 milligrams per liter.

FILLED CHEESE.

On the authority of the Produce Exchange Bulletin, I give the following account of the manufacture of filled cheese :

The process consists in taking all the cream out of the milk by the separator, and then taking the skim milk up and charging the vat just before it is set with deodorized lard, cotton-seed oil, or other fat. The oil is taken up in the curd and mechanically held there, the cheese curd simply being used as a capsule in which to carry it. There is no assimilation or chemical affinity between the curd and its contents.

Prof. H. A. Weber, of Columbus, Ohio, has made comparative analyses of samples of genuine and artificial cheese, with the following results:

	Genuine cheese.	Artificial cheese.
	<i>Per cent.</i>	<i>Per cent.</i>
Water	35.42	52.73
Ash	2.47	2.69
Fat	34.66	2.63
Caseine, sugar, &c	30.45	41.95
Total	103.00	100.00

¹Pharm. Zeitung, vol. 32, p. 59.

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U. S. DEPARTMENT OF AGRICULTURE.

DIVISION OF CHEMISTRY.

BULLETIN

No. 13.

FOODS

AND

FOOD ADULTERANTS.

BY DIRECTION OF

THE COMMISSIONER OF AGRICULTURE.

PART SECOND:

SPICES AND CONDIMENTS.

BY

CLIFFORD RICHARDSON.

WASHINGTON:
GOVERNMENT PRINTING OFFICE.

1887.



[BULLETIN No. 13.]

PART 2.—SPICES AND CONDIMENTS.

U. S. DEPARTMENT OF AGRICULTURE,
DIVISION OF CHEMISTRY,
Washington, D. C., March 15, 1887.

SIR: Having been directed by you to make an examination of the adulteration of spices and condiments, I have been engaged at different times during the past year upon the subject, and with some aid from the assistants in the laboratory in performing the chemical determinations have prepared the following report, which forms part 2 of Bulletin No. 13 of this Division. It has been my endeavor to collect all the available information in regard to the extent and character of this adulteration in all parts of the world, and to describe for the benefit of investigators the means to be employed for its detection. More particularly I have entered into an examination of the spices and condiments found in the markets of Washington and Baltimore, and have tested with them the methods of investigation which are described.

It has seemed appropriate to divide the report into two parts, the first being devoted to a popular exposition of the origin of the spices and the means employed for their adulteration, while the second contains a more extended technical discussion of the subject and of the results of analysis, both microscopical and chemical, and the methods which have been proposed by recent authorities for the detection of sophistication. I have been assisted in the chemical work by Messrs. Knorr, Trescot, and Fake, assistants in the laboratory of the Division, who have completed a large portion of the determinations under my supervision, and I am indebted to Dr. Battershall, of New York, for the privilege of examining the advanced sheets of his work on food adulteration.

To the standard authorities, Hassall, Koenig, Schimper, and others, I owe much for the information derived from them, although their work is not entirely applicable in this country, and I have copied from Schimper some of his diagramatic drawings of the spices, which, although extremely deceptive to the beginner, may in their proper way be made of value for reference. Other sources of information I have acknowledged in the body of the report.

The amount of adulteration which has been detected is extremely large and of a nature which apparently arouses but little prejudice on the part of the consumer, being free from associations of uncleanness. Could only a portion of the unfortunate dislike for oleomargarine be directed toward the spices, the result would be that much wasted energy would be turned into a profitable channel. The necessity for some means for the suppression of the present universal sophistication of spices and condiments seems urgent.

Very respectfully,

CLIFFORD RICHARDSON.

Dr. H. W. WILEY.

SPICES AND CONDIMENTS.

PART I.

Under this head are included substances which while not in themselves foods serve to render the latter more palatable, and to stimulate digestion. They occupy an important position in the diet of the human race, and are largely subject to adulteration or sophistication. Among the more common are: Peppers of various kinds, mustard, cloves, cinnamon, cassia, allspice or pimento, nutmeg, mace, ginger.

These substances are so often and seriously adulterated because it is readily accomplished, owing to the custom of putting them on the market in a ground condition, which prevents the recognition of quality from mere appearance, and because so many cheap substitutes or diluents are readily found which resemble the real article. In addition, the demand on the part of the poorer classes for the cheapest possible supply and the competition brought about by this demand in the trade has, owing to general high prices attached to most of the spices of good quality, fostered and extended the practice.

At the present time in several of our largest cities the price to be paid for a spice is named by the retail dealer, and he is then furnished from the spice-mill with a mixture containing the largest amount of pure material which can be supplied for the money, the necessary weight being made up of diluents of some cheap but harmless substance, different grades being distinguished by one firm as pure, extra, No. 1, and superior, none of which are pure and many of which are mere variation in labels with none in quality. As examples, the fact that a New York firm, it is understood, in a short time used and put upon the market in their spices more than 5,000 pounds of cocoanut shells, and the following quotations from a journal devoted to spice milling show how universal and open the custom has become. On a prominent page is found :

“All necessary information for spice manufacturing supplied.”

Attention should be paid to the use of the word manufacturing.

Then the following advertisements appear, from which the names have been stricken out:

MANUFACTURER OF
SPICES, SPICE MIXTURES, AND MUSTARDS,

181 ——— *Street, N. Y.*

Goods made to order for Wholesale Grocers and Druggists; also, Grinding
done for Jobbers who pack their own Goods.

Spice Mixtures and Cayenne Pepper a Specialty.

17 ——— STREET, NEW YORK,
MANUFACTURER OF
ALL KINDS OF SPICE MIXTURES.

My celebrated brand of P. D. Pepper is superior to any made.

Samples sent on application. Goods shipped to all parts of the United States. Spice mixtures a specialty. Spices ground for the trade. We are the Inventors of Suction Coolers.

The firms advertising do not hesitate to call themselves manufacturers instead of millers.

It is easy to see how difficult it must be to bring this state of things to an end without some governmental action, it being improbable that by any means of agreement among themselves the grinders of spices could unite in doing away with the practice, or that any education of the masses will teach them to refuse to purchase a ground spice at a price which is far below that of the unground article. This alone, the relation between the prices of ground and unground spices, is often sufficient to point out the fact that a ground spice must be largely diluted, and, on the other hand, when purchasing from a reliable dealer, a slight increase in cost over that of the spice in its original form is fair evidence of the purity of the powder. Those who desire pure ground spices can almost always obtain them by paying their value. They are by no means uncommon in the market, but as long as there are those who do not know that it is for their interest to buy the best, rather than a cheap article on account of its low price, such people must suffer or be protected by legal enactments which shall prevent and prohibit the existence of such mixtures. Until this is done the supply of a demand which certainly exists may be considered to be, at the least, justifiable on the part of the spice millers, and education of those ignorant of the state of the trade must be the preliminary to legislation upon the subject. When proper legislation has found a place on the statute-books the manufacturers will find themselves in a position where, without detriment to themselves, they can all unite in giving up the practice. Under

the laws for the prevention of the adulteration of foods which have been in operation in Germany, England, France, Canada, and a few of our States, during a longer or shorter period of time, a large share of attention has been given to the adulteration of spices and condiments and the means of detecting them. Fortunately the latter are not difficult, and the results have been an awakening of the communities in these countries to an appreciation of the advantages of pure spices and the placing of the method of detection on a more certain basis.

EXPERIENCE IN COUNTRIES HAVING PUBLIC ANALYSTS.

In England the public mind had been so far educated by the publications of private investigators, such as Hassall, that in 1860 laws were passed for the prevention of the adulteration of food and drink. These have been modified and repealed, so that the present law dates from 1875 and amendments of 1879. Unfortunately there is no Government report, of which we are aware, upon the results of the scientific work done by those employed under the act, and we are indebted to the Society of Public Analysts for a large portion of the information which is at our disposal in regard to adulteration in England. We have also in the publications of Hassall, Blyth, and Allen volumes which give the most recent scientific data as to the best methods for the detection of adulteration, and illustrations of the forms in which foreign matter occurs. On Dr. Hassall's work is founded many of our present methods of examining foods microscopically, and especially spices and condiments. In the Analyst, the publication of the Society of Public Analysts, will be found among the proceedings of the society, in papers of individuals, and in reports of prosecutions, much information in regard to the status of adulteration in England during the last eleven years, including the material used for adulteration of spices and the means of detecting it. The lack of an official publication of the results of all that has been done in regard to particular samples and of the methods employed for their examination is, however, much to be regretted. There is the same difficulty in Germany. The law of the Empire of 1881 provides for the prevention of the adulteration of the substances which we have under consideration, but no reports on the execution of the law or of the results, scientific or otherwise, have been made available to us. Much, however, has been published in the German technical and scientific journals on the methods of detecting adulterants, which is of the greatest value.

In France, the laboratory of the prefecture of police of Paris, which has control of the investigation of the food supplies of that city, makes an elaborate report annually, of which, however, but a small portion is devoted to spices, although they are recognized as being largely adulterated, pepper, for example, being mixed to an astonishing extent with ground olive stones. Other cities of France have municipal laboratories whose reports, if any there are, have not reached us.

In this country Canada makes a much better statement of the results which have followed the enforcement of the adulteration-of-food act of 1876 than is done anywhere else. The commissioner of inland revenue has published annually for eight or ten years a statement showing the entire number of samples analyzed, the persons supplying them, and their composition and adulterants. Spices occupy a prominent position in the reports, and a collation of the results of the investigations of the several public analysts is of interest. In 1878, when the reports first become available to us for reference, the summary of the spices analyzed showed:

Articles.	Unadulterated.	Adulterated.	Per cent. adulterated.
Allspice.....	1	11	92.5
Cassia.....	2	2	50.0
Cloves.....	3	15	83.3
Cinnamon.....	3	6	66.6
Ginger.....	5	8	61.5
Mustard.....	38	100.0
Pepper.....	12	28	70.0

This enormous amount of adulteration, amounting to nearly a universal custom, was followed in 1879 by a similar report:

Articles.	Unadulterated.	Adulterated.	Per cent. adulterated.
Allspice.....	6	10	62.5
Cassia.....	1
Cloves.....	7	9	56.2
Cinnamon.....	3	16	84.2
Ginger.....	5	6	55.5
Mace.....	5	1	16.6
Mustard.....	3
Pepper.....	22	21	48.8

In 1880 there were reported as the results of the public analysts' work:

Articles.	Unadulterated.	Adulterated.	Per cent. adulterated.
Allspice.....	15	6	31.6
Cloves.....	12	10	45.5
Cinnamon.....	6	16	72.7
Ginger.....	9	8	47.1
Mustard.....	16	100.0
Pepper.....	24	18	42.9

There is a very slight improvement apparent, but it must be remembered that in examinations of this description the specimens selected are always of a suspicious nature, those which are already known to be pure being omitted, so that year by year the list of brands which are excluded from examination increases.

In 1881 and the following years results were tabulated as follows:

Articles.	1881.		1882.		1883.		1884.		1885.	
	Gen- uine.	Adul- ter- ated.	Gen- uine.	Adul- ter- ated.	Gen- uine.	Adul- ter- ated.	Gen- uine.	Adul- ter- ated.	Gen- uine.	Adul- ter- ated.
Allspice	8	17
Cloves	6	7	12	10
Cinnamon or cassia....	12	12
Ginger.....	7	5	7	16	37	29
Mace	1	1
Mustard.....	2	2	11	*39
Nutmeg	1
Pepper	16	29	14	28	31	29
Cayenne	13	11
Total	53	73	34	32	48	86	38	112	103	118

* Many labeled mixtures.

It is seen that several years after the enactment of the law adulteration of spices is as enormous as at first. This, however, seems to arise largely from lack of prosecution and non-enforcement of the law. Of the occurrence of adulterants in spices, the chief analyst says, in his report for 1885:

During the year considerable attention was paid to spices and condiments. Viewing the fact that in the past a very large amount of adulteration had been reported as prevailing in these substances, and with a view to ascertaining whether the adulteration was practiced by the manufacturers or by the dealers, a systematic visitation was made of all the spice grinders in the Dominion (or of all that could be recognized as such), and their factories and stores were inspected under sections 7 and 8 with the results as shown in the appendix.

The examination of nineteen samples of ground cinnamon resulted in finding seven genuine; four to consist of a substitution of cassia; one was adulterated with cassia, and six with other inert matter; one consisted of cassia adulterated with foreign vegetable matter.

Of ground cloves, twenty-two samples were examined. Twelve proved to be pure and ten adulterated, the adulterant chiefly used being clove stems, pea-meal, roasted and ground cocoanut shells.

Of sixty-six samples of ground ginger, twenty-nine were reported as being adulterated, almost exclusively with wheat flour, non-injurious to health, doubtless; but unless the purchaser be duly warned of the nature of the compound his pocket would be seriously prejudiced, if not injured, as this sophistication was practiced to the extent of from 10 to 15 to from 25 to 40 per cent., the pungency being imparted by the judicious admixture of cayenne pepper.

Fifty samples of mustard were examined, and many of these were properly sold as "compound" or mixtures, but one of the worst samples was sold with a label guaranteeing it to be "ground from finest English seed, and free from adulteration." Of the fifty samples, nine were reported genuine, two of excellent quality, and thirty-nine were all, more or less, admixtures of mustard seed or mustard cake (from which the natural fixed oil had been expressed) with wheat flour and turmeric, and, in some cases, with corn-starch or bean meal, in varying proportions, up to as high as 50 or even 60 per cent. It was formerly contended that the addition of wheat flour or other inert matter was a necessity, to give the ground mustard keeping qualities and make the condiment palatable by softening its natural acidity. But the most reputable manufacturers have demonstrated the fallacy of this contention by the produc-

tion of an absolutely pure mustard, which has received public acceptance and appreciation, and two, at least, of our home manufacturers are happily following in their steps. It is a question yet to be decided how far the use of mustard cake, deprived of the natural fixed oil, is permissible in the manufacture of this condiment. Dr. Ellis' observations on this matter are very much to the point, and have received confirmation by similar experiments in my laboratory, and doubtless when next the analysts meet in conference this question will be settled in a manner favorable to the use of mustard cake.

Twenty-four samples of cayenne pepper were examined, of which fourteen were reported adulterated, but three of these were appealed to the judgment of the chief analyst, and the decision of the public analysts was not sustained, as will be seen on reference to "appeal cases." The remaining ten were reported adulterated with wheat flour and colored earth, in one case to the extent of 50 per cent. The other ten samples were reported unadulterated, save one, which was doubtful, it apparently having been artificially dressed with a fixed oil.

Sixty samples of ground pepper, black and white, were examined, of which thirty-one are reported as unadulterated, one doubtful, and twenty-eight all more or less adulterated—the generality of them to the extent of from 10 per cent. to 20 per cent., but the more flagrant cases from 30 up to even 75 per cent. in one case. The adulterant is chiefly farinaceous matter, also mustard husk, pepper hulls, clay, sand, and, not the least conspicuous, *ground cocoonut shells*—doubtless an innocent admixture, so far as health is concerned, but decidedly not a material of a character to improve the flavor or value of the pepper as a condiment.

As stated, these samples of spices were all obtained from either the actual producer or wholesale distributors; and the results prove that whether or not the retail vender still further "improves" his spices, &c., before retailing them, his demand for a cheap, adulterated article is amply provided for by the manufacturing dealer.

For the most part, the producers of these sophisticated goods expressed themselves anxious for the enforcement of the law, that, if sold, they should be distinctly labeled as impure. Some, on the other hand, contended that the public was benefited by a slight admixture—that a really better article could be supplied at a lower price if the finest and freshest spices were ground with an admixture of inert matter, than a thoroughly pure article, but ground from old or perished spices—a specious contention, utterly untenable in the true interests of the public.

But have not the producers of these sophistications some justification; is not the supply of a demand which, undoubtedly, has existed, a justifiable enterprise, whatever that demand may be, so long as it is within the law? Ignorance does, undoubtedly, demand cheapness, and a demand thus ignorantly made is only too surely supplied, and hence the need for costly legislation to protect an ignorant and thoughtless public against itself, for it does demand the very goods which the analyst must condemn and the vender be prosecuted and fined for selling, whereas the public's reckless ignorance is the chief cause, and should suffer some measure of the penalty. It is time that through the operation of this act such ignorance should be cleared away, and the public be enlightened and awakened to its own true interests.

These remarks apply equally well to much of the spices sold in the United States. Massachusetts, New York, New Jersey, and Michigan alone have laws* of any value in regard to the adulteration of food, and it is easy to see in what condition the spices and condiments sold in other parts of the country must be. In Massachusetts, where investigations under the law have been going on since 1882, it has been shown, as in other localities, that the adulterants of spices are numerous but harmless. Dr. Sharpless, in his report of 1882 upon the subject, remarks,

* See Appendix.

that he agrees with the opinion expressed by Dr. Leeds, of New Jersey, in his report of 1880 to the New Jersey State board of health, that there has been much sensational writing upon the subject. This is perhaps the case with some few writers of that stamp, but it can have done no harm, for it has not produced sufficient effect upon the public to create a demand for any purer spices, as appears from the figures of Dr. E. S. Wood in 1882, who reports in regard to the samples he examined as analyst of foods for Massachusetts :

Articles.	Genuine.	Adulterated.	Per cent. adulterated.
Black pepper.....	20	44	68.75
White pepper.....	13	31	70.45
Red pepper.....	5
Mustard.....	29	47	61.84
Ground cloves.....	0	11	100.00
Cassia.....	0	2	100.00
Pimento.....	2
Ginger.....	1

This is nearly as serious a condition as was found in Canada, but again, in 1885, Dr. Wood reports :

Articles.	Genuine.	Adulterated.	Compounds.	Per cent. adulterated.
Mustard.....	31	37	20	64.8
Cloves.....	15	76	83.5
Cassia and cinnamon..	48	26	1	36.0
Ginger.....	55	17	23.6
Allspice.....	30	8	21.1
Ground mace.....	9	9	50.0
Black pepper.....	6	19	3	78.6
White pepper.....	21	39	65.0

The above is little or no improvement. He found the common adulterants of mustard to be flour, turmeric, and sometimes a little cayenne. Cloves suffered from extraction of the volatile oil and the addition of clove stems, allspice, burnt shells, and other cheap substances. Cassia contained ground shells and crackers; ginger was in many cases colored, and in some instances wheat and corn flour and clove stems were present. Allspice is too cheap to be often adulterated, but in eight samples mustard hulls, ground shells, clove stems, and cracker dust were found. In mace, flour and cornmeal were diluents, and for the peppers, crackers, mustard hulls, pepper dirt, powdered charcoal, rice, corn, and buckwheat.

Under the New York law of 1881, Prof. S. A. Lattimore investigated a number of spices and spice mixtures submitted to him.

Professor Lattimore's report furnishes abundant proof in support of the common impression regarding the adulteration of spices.

On the results of his examination of the commercial ground spices he comments, after giving the proportions which were found adulterated, in these words:

Articles.	Samples examined.	Adulterated.	Percentage of adulteration.
Mustard	18	12	66.6
Ginger	15	9	60.0
Allspice	27	19	70.4
Cinnamon	22	18	81.8
Cassia	7	4	57.1
Cloves	21	16	76.2
Pepper:			
Black	40	28	70.0
White	7	5	71.4
Red	10	5	50.0
Mace	8	4	50.0
Nutmeg	5	2	40.0

As the above table shows, a large proportion of them are adulterated, and that with substances presenting a certain uniformity. The spices present an inviting field for the exercise of fraudulent arts. They are almost universally sold in the form of fine powder and in opaque packages, which do not admit of easy examination on the part of the purchaser. Consequently any cheap substance which may be easily pulverized to a similar degree of fineness, and which possesses little distinctive taste or odor of its own, answers the purpose; so that the list of adulterants for this class of articles is naturally very large. The adulterations found in the samples now under consideration may be classed into four groups. First, integuments of grains of seeds, such as bran of wheat and buckwheat, hulls of mustard seed, flax seed, &c. Second, farinaceous substances of low price, such as are damaged by the accidents of transportation or long storage—such as middlings of various kinds, corn-meal, and stale ship's bread. Third, leguminous seeds, as peas and beans, which contribute largely to the profit of the spice mixer. Fourth, various articles chosen with reference to their suitability for bringing up the mixture as nearly as possible to the required standard of color of the genuine article. Various shades, from light colors to dark browns, may be obtained by the skillful roasting of farinaceous and leguminous substances. A little turmeric goes a great way in imparting the rich yellow hue of real mustard to a pale counterfeit of wheat flour and terra alba, or the defective paleness of artificial black pepper is brought up to the desired tone by the judicious sifting in of a little finely pulverized charcoal. Enough has been already given to show that the field for sophistications of this sort is a wide one, and offers large scope for the development of inventive genius; so that each manufacturer of articles of this class would be likely to possess his own trade secrets. It will be observed that the adulterating materials just mentioned all belong to the class claimed to be harmless. In no instance has any poisonous substance been discovered. The proportion of foreign and genuine substances in the spices varies between wide limits, in some instances the former being slight; in others, the latter seemingly present in just sufficient quantity to impart faintly the requisite taste or odor. Even this small proportion of the professed article is occasionally further diminished by the substitution of other substances; as, for example, in imparting to corn-meal finely ground a pungency suggested by real ginger by the addition of a little salt and red pepper.

It is probably not so widely known as it should be that the demand for the materials for adulteration has called into existence a branch of manufacturing industry of no insignificant magnitude, having for its sole object the production of articles known as "spice mixtures" or "pepper dust." The use of "pepper dust," or, as the article is commonly designated in the technical language of the trade by its abbreviation "P. D.," is a venerable fraud.

The manufacture of "P. D." is now a regular branch of business, and the original and specific term "pepper dust" has expanded with the progress of inventive art to generic proportions, until now we have as well-known articles sold by the barrel "P. D. pepper," "P. D. ginger," "P. D. cloves," and so on through the whole aromatic list. When it is considered that these imitations, lacking only such flavoring with the genuine article as the dealer thinks necessary to make his goods sell, are sold at from three to four cents a pound, and the retail price paid by the consumer is compared with it, the strength of the temptation to engage in such practices is clearly seen. When manufacturers openly advertise themselves as assorters and renovators of merchandise, and openly propose to cleanse musty and damaged beans by a new and patented process, it is full time that its significance should be considered by the public.

From the investigations, which have been quoted, it appears that the adulterants which are met with in this country are very numerous. Under the head of spice mixtures, or "P. D.," much refuse of all descriptions is used up, and there are such changes in the character of the material from time to time as the sources of damaged material or refuse at hand may suggest. The diluents used in Baltimore and in the District of Columbia seem to be different from those in New York, and, in consequence, some of the adulterants which are mentioned most commonly in the reports from the North are not found in the spices of southern millers. While it is possible, therefore, to give a list of substances which have been used as adulterants, it is quite out of the question to say in what directions the ingenuity of spice-mixers will extend in the future. The following contaminations in the various spices have been already noted in this country :

Spices.	Adulterants.
Allspice	Spent cloves, clove stems, cracker dust, <i>ground shells</i> or charcoal, <i>mineral color yellow corn</i> .
Cayenne	<i>Rice flour</i> , salt and ship-stuff, <i>yellow corn</i> , <i>turmeric</i> , and <i>mineral red</i> .
Cassia	<i>Ground shells</i> and <i>crackers</i> , <i>turmeric</i> , <i>minerals</i> .
Cinnamon	<i>Cassia</i> , peas, starch, <i>mustard hulls</i> , <i>turmeric</i> , <i>minerals</i> , <i>cracker dust</i> , <i>burnt shells</i> or charcoal.
Cloves	<i>Spent cloves</i> , <i>clove stems</i> , <i>minerals</i> , allspice, roasted shells, wheat flour, peas.
Ginger	<i>Cereals</i> , <i>turmeric</i> , <i>mustard hulls</i> , <i>cayenne</i> , peas.
Mace	Cereals or starch, buckwheat, <i>wild mace</i> .
Nutmeg	Cereals or starch, wild nutmeg.
Pepper	<i>Refuse of all sorts</i> , <i>pepper dust</i> , <i>ground crackers</i> , or <i>ship stuff</i> ; <i>rice</i> , <i>mustard hulls</i> , <i>charcoal</i> , <i>cocoanut shells</i> , <i>cayenne</i> , <i>beans</i> , <i>bran</i> , <i>yellow corn</i> .
Mustard	<i>Cereals</i> and starch, <i>turmeric</i> , peas, <i>yellow corn meal</i> , <i>ginger</i> , <i>gypsum</i> .

The materials in italics have been identified in spices examined in the laboratory of this division, but some of the commoner adulterants have not been found. Considering the spices individually, there are certain peculiarities, as they are met with pure and in the trade, which should not be overlooked.

MUSTARD.

Mustard, as sold in the ground state, should be the farina or flour of the black or white mustard seed—that is to say, the flour from the interior of the seed bolted or separated from the hulls. The two kinds of

seed, although derived from plants of the same genus, are somewhat different in their chemical composition. The black seed is much the most pungent, and develops, on mixing with water, a volatile oil, which gives this condiment its penetrating character. There is also present in the seed a complicated organic substance of a bitter nature, to which is due some of the peculiar flavor, and while the white seed forms no volatile oil with water, it contains more of the bitter substance. It is, therefore, very common to mix the two in grinding. The sources of the seed are various. In our markets at present there are quoted California black and white, Dutch, Trieste black, and English—the last being the most valuable.

In the manufacture of the seed into flour for the market, two customs have arisen which change the nature of the original substance, and therefore would commonly come under the head of adulteration. One is extremely old, the addition of wheat flour for the purpose of making the condiment keep better. This necessitates the restoration of the yellow color by turmeric, or other dye-stuff. These diluents are harmless as a rule, but there seems to be no reasons for their use, and it is gradually becoming commoner to find mustard free from them in English brands.

The other custom is the abstraction of the fixed oil by pressure before grinding the seed. The percentage of this oil is over 30. It adds nothing to the flavor of the mustard, probably injures its keeping qualities, makes the seed more difficult to mill, and its removal is therefore a benefit. It is a nearly universal custom at the present day in this country, and is not considered as fraudulent by the Canadian analysts.

Falsifications of mustard other than those mentioned are not common, although gypsum has been found in low-grade mustard and several other adulterants, among them ginger of low grade. The hulls bolted from the flour in the process of manufacture are preserved and form the basis of the adulteration of many other spices.

PEPPER, BLACK AND WHITE.

Pepper is more in demand than any other spice, and in consequence is more adulterated. Its appearance in the ground form, especially of the black, is such as to make it possible to use all sorts of refuse for this purpose, and almost everything that has been used as an adulterant has been found in pepper. White pepper, which is simply the black deprived of its outer black coats, is, of course, less easily falsified; but in France is diluted to an immense extent with ground olive stones, which bear a striking resemblance. Among the samples from Washington grocers, pepper sweepings—that is, husks and dirt—rice or corn, and mustard-hulls were the commonest admixtures. Sand is said to be very commonly added abroad, but has not been met with here. In Canada and New York ground cocoanut-shells are a cheap source of adulteration, but they have not extended so far south.

Specimens from Baltimore mills of very low-quality goods were found to contain but little pepper, and that of the worst quality, being made up of cracker dust, yellow corn, cayenne, and charcoal in so disgraceful a way as to be visible to the unassisted eye on close examination.

The quality of a ground pepper can be told by an expert from its weight and color, and on examination with a lens of low magnifying power. The particles are not coarsely ground, and it is not difficult to pick out pieces of husk, yellow corn, and rice, and, if necessary, a more careful investigation under a microscope of higher power will serve for confirmation. Black pepper, in our experience, is much more liable to adulteration than white, although it is perfectly easy to dilute the latter with broken rice or cracker dust, which are inexpensive. All these materials, fortunately, owing to the grossness of the adulteration, are readily recognized, and there is hardly the necessity for recourse to chemical analysis. There has been, however, considerable investigation in this direction, so that there are means of confirming the optical examination which are of great value. Determination of the amount of starch is one of the methods upon which some reliance can be placed; for, if under the microscope foreign starch is not detected, then the addition of "P. D." or other starch free adulterants, will diminish the percentage found, and the reverse. In this way, too, one is able to arrive at an approximate conclusion as to the proportion of adulterants added, which can only be estimated within wide limits under the microscope.

In spite of the immense amount of adulteration, it is possible, from the best shops, to obtain pure ground peppers, but it is at the same time safer with a family spice-mill to grind the whole berries as they are needed. The sources of our pepper supply are Tellicherry on the west coast of Hindostan, which is graded high, and Penang and Singapore for the east, Sumatra, Java, &c. The importations are principally through London, and not direct. The supply of ground pepper from England will usually be found more pure than our own brands, and at the same time is naturally more expensive.

CAYENNE OR RED PEPPER.

This condiment should consist of the ground pods of any of several species of *Capsicum*, known as chilies or peppers. It is said to have been adulterated with many substances, brick dust, red lead, and coloring matters; but this has only been found to be the case in two cheap Baltimore cayennes, while in Washington only rice has been detected, but that quite frequently. Inferior material is no doubt often ground, but the small value of the pods and the small quantity consumed does not tend to increase adulteration.

GINGER.

Ginger is the root, or, technically, rhizome, of a plant somewhat similar in appearance to our iris and flag. It is grown in various parts of the world and prepared with great care and great carelessness, being at

times scraped and bleached, at others simply dried in any condition, so that there is an immense number of varieties and qualities to be found in the market. They all, however, retain sufficiently the marked peculiarities of the starchy fibrous root to make the detection of adulterants easy. The common ones are the addition of wheat flour or some starch as a diluent, the coloring with turmeric to suit a popular fancy for gingerbread or of spent material which has been used in making tincture. Mustard hulls and cayenne are also found in some cases, but have seldom been detected here. They are added to give pungency and make up for the addition of flour. Their detection is easy. The sources of our supply are Jamaica and the West Indies, Cochin China, Africa and India. Jamaica is the best and most carefully prepared.

CLOVES.

The flower buds of the clove tree carefully picked and dried constitute the spice known by that name. Their valuable properties are due to the volatile oil which they contain, the best having as much as 16 per cent.

The removal of this oil is so very easy that it is the commonest method of deception to do so before grinding the spice and to then dispose of it as pure. We have ready means of determining the loss chemically, but the microscope gives no indications. The addition of the cheaper clove stems is also practiced, as they cost but 6 cents when the buds cost 27. The microscope reveals their presence by certain cells which they contain which are absent in the buds.

Pimento is sometimes substituted in part or entirely, as it has a clove-like flavor but only 4 or 5 per cent. of volatile oil. It is worth less than one-fifth the price of cloves. Its chemical composition and its structure, that of a berry, reveals its presence. The addition of the coarser adulterants, mineral matter, cocoanut shells, flour, peas, and the like, have only been observed in two instances, but no doubt frequently occur, as has been found in Canada.

The sources of our supply are the East Indies (Amboyna), African (Zanzibar), and American, ranking in value in the order named. Cloves should, if possible, be always purchased whole, as they deteriorate less readily in that form.

CINNAMON AND CASSIA.

These spices are the barks of several species of the genus *Cinnamomum*, the true cinnamon being a native of Ceylon, where it is largely cultivated, and the cassias being derived from several other species growing in China, India, and the East Indies. Cinnamon as it reaches the market is very thin, the outer and inner coats of the bark having been removed. Cassia on the other hand is thick, as it consists of the entire bark and can be distinguished by its retaining its natural outer surface. Cinnamon is by far more valuable than the cassia, as there is a smaller supply and intrinsically it contains a much greater propor-

tion of volatile oil and that of higher and more delicate aroma. In consequence cassia is largely substituted for cinnamon, and in fact not a particle of ground cinnamon can be found in the market. It can be found in the whole condition in good quality only in drug stores. Cassia exists in many forms and qualities, and sells at wholesale at from 7 to 40 cents a pound. That known as Saigon is the best, and that exported from Batavia the poorest. Cassia buds also hold a small place in the market.

The detection of the substitution of cassia for cinnamon, since the barks are of trees of the same species, is more difficult than is usually the case and may prove troublesome to a novice. The presence of more woody fiber in the latter with the aid of chemical analysis serves, however, as a reliable distinction. In the samples which have come into our hands not a particle of material labeled ground cinnamon proved to be anything other than cassia. The spice millers appeared, however, to be satisfied to stop at this point and in only one case was there addition of cheap stuff to the cassia. When added there is no difficulty in detecting it as has been done here and in Canada, where peas, starch, ground shells, and crackers have been found in powder labeled both cassia and cinnamon.

The barks can, in most cases, and especially the cinnamon, be used nearly as well in the whole condition and should at least be so purchased and then ground. A slight acquaintance with the appearance of the different qualities will teach one the proper selection to make.

NUTMEG AND MACE.

These spices are different portions of the fruit of a tree known as the nutmeg tree, *Myristica fragrans*, the nutmeg being the inner kernel and the mace one of the outer coats or arillus. The tree grows principally on the Banda Islands and the spices reach us through London. They can always be obtained in their original condition and should be so purchased. When ground they are mixed with diluents of various descriptions, principally cereals and their refuse, which are easily detected.

Owing to the infrequency of the sale of the powdered nutmeg and mace, their adulteration has attracted but little attention.

SOURCES OF OUR SPICE SUPPLY.

Although the countries where the spices are grown have been already given in a general way and in a later chapter will be given more in detail, these countries, in many instances, are not the direct sources of our supplies. For instance, of the pepper imported in the fiscal year ending June 30, 1886, amounting to nearly 12,000,000 pounds, over 8,000,000 came to us from England, and about 3,000,000 from the British East Indies, including the ports of Singapore and Penang. In regard to the other spices data are found in the annual statement of the Bureau

of Statistics of the Treasury Department and in some additional tables furnished to this office by the Bureau, which are here given:

Imports of unground spices, free of duty, for the year ending June 30, 1886.

Countries from which imported.	Nutmegs.		Pepper.		All other.	
	Quantity.	Value.	Quantity.	Value.	Quantity.	Value.
	Pounds.		Pounds.		Pounds.	
Argentine Republic						
Austria			2, 128	\$225		
Belgium						
Brazil					194	\$32
Central American States:						
Costa Rica						
Guatemala			12	1		
Honduras						
Nicaragua						
San Salvador						
Chili			5, 905	498		
China	11, 825	\$3, 982	344, 552	45, 808	257, 402	21, 737
Denmark						
Danish West Indies						
Greenland, Iceland, and the Faroe Islands						
France						
French West Indies	300	120				
French Guiana						
Miquelon, Langley, and Saint Pierre Islands						
French Possessions in Africa and adjacent islands						
French Possessions, all other						
Germany	4, 740	2, 293			16, 831	1, 832
England	247, 839	98, 412	8, 214, 037	1, 157, 264	2, 003, 255	176, 535
Scotland						
Ireland						
Gibraltar						
Nova Scotia, New Brunswick, and Prince Edward Island						
Quebec, Ontario, Manitoba, and the Northwest Territory			1, 465	338	740	35
British Columbia						
Newfoundland and Labrador						
British West Indies	3, 344	889	480	9	3, 017, 943	146, 880
British Guiana						
British Honduras						
British East Indies	464, 862	179, 169	2, 927, 472	403, 090	1, 006, 938	66, 536
Kong-Kong					1, 089, 089	91, 650
British Possessions in Africa and adjacent islands			11, 984	1, 042	1, 563, 073	59, 101
British Possessions in Australasia						
British Possessions, all other			840	66		
Greece						
Hawaiian Islands						
Hayti						
Italy						
Japan						
Liberia					4, 233	296
Mexico	50	37	48, 255	7, 528	907	100
Netherlands	210, 635	83, 412			31, 038	7, 292
Dutch West Indies						
Dutch Guiana						
Dutch East Indies	245, 912	90, 065	3, 261	360	817, 756	35, 618
Peru						
Portugal						
Azore, Madeira, and Cape Verde Islands						
Portuguese Possessions in Africa and adjacent islands						
Roumania						
Spain					151	36
Cuba					856	241
Porto Rico					101, 338	4, 302
Sweden and Norway						
Switzerland						
Turkey in Europe						
Turkey in Asia						
Venezuela					1, 160	89
All other countries and ports in Asia						
All other countries and ports in Africa			283, 062	28, 154	851, 307	66, 624
Total	1, 189, 507	458, 379	11, 843, 453	1, 644, 383	10, 767, 211	678, 936

Imports of ground spices, dutiable, for the year ending June 30, 1886.

Countries from which im- ported.	Quantity.	Value.	Countries from which im- ported.	Quantity.	Value.
	<i>Pounds.</i>			<i>Pounds.</i>	
Argentine Republic			British East Indies		
Austria	991	\$225	Hong-Kong	114	\$8
Belgium			British Possessions in Africa and adjacent islands.		
Brazil			British Possessions in Aus- traliasia.		
Central American States:			British Possessions, all other.		
Costa Rica			Greece		
Guatemala			Hawaiian Islands		
Honduras			Hayti		
Nicaragua			Italy	22	2
San Salvador			Japan		
Chili			Liberia		
China	1	1	Mexico	448	72
Denmark	50	8	Netherlands	3,312	246
Danish West Indies			Dutch West Indies		
Greenland, Iceland, and the Faroe Islands			Dutch East Indies		
France	3,028	913	Peru		
French West Indies			Azores, Madeira, and Cape Verde Islands		
French Guiana			Russia on the Baltic and White Seas	1,101	93
Miquelon, Langley, and Saint Pierre Island			San Domingo		
French Possessions in Africa and adjacent islands			Spain	35,442	3,087
French Possessions, all other			Cuba	2,734	351
Germany	556	103	Porto Rico		
England	644,022	164,687	Philippine Islands		
Scotland			Sweden and Norway		
Ireland	1,758	611	Switzerland		
Gibraltar			Turkey in Europe		
Nova Scotia, New Brunswick, and Prince Edward Island	5	1	Turkey in Asia		
Quebec, Ontario, Manitoba, and the Northwest Terri- tory	115	15	United States of Colombia		
British Columbia			Uruguay		
Newfoundland and Labrador			Venezuela		
British West Indies			All other countries and ports in Africa		
British Guiana					
British Honduras			Total	693,699	170,423

*Statement showing, by customs districts and ports the quantities and values of spices im-
ported and entered for consumption in the United States during the year ending June 30,
1886.*

FREE OF DUTY.

Customs districts and ports.	Cassia buds.		Cassia, cassia vera.		Cinnamon, and chips of	
	Quantity.	Value.	Quantity.	Value.	Quantity.	Value.
	<i>Pounds.</i>		<i>Pounds.</i>		<i>Pounds.</i>	
Aroostook, Me						
Baltimore, Md.					12,127	\$824.00
Boston, Mass.						
Brazos de Santiago, Tex.						
Chicago, Ill.						
Cincinnati, Ohio						
Corpus Christi, Tex.						
Detroit, Mich.						
Duluth, Minn.						
Galveston, Tex.						
Key West, Fla.					2	.50
Middletown, Conn.						
Minnesota, Minn.						
New Orleans, La.						
New York, N. Y.	232,718	\$14,496	1,838,649	114,681	64,555	10,274.00
Oregon, Oreg.						
Oswegatchie, N. Y.						
Paso del Norte, Tex.						
Philadelphia, Pa.	5,313	566	12,451	978		
Saluria, Tex.						
San Francisco, Cal.						
Saint Louis, Mo.					2,243	387.00
Willamette, Oreg.						
Total	238,031	15,062	1,851,100	115,659	78,927	11,485.50

Statement showing, by customs districts and ports, the quantities and values of spices imported and entered for consumption, &c.—Continued.

FREE OF DUTY.

Customs districts and ports.	Cloves.		Clove stem.		Ginger root.	
	Quantity.	Value.	Quantity.	Value.	Quantity.	Value.
	Pounds.		Pounds.		Pounds.	
Aroostook, Me						
Baltimore, Md.	11,098	\$1,500.00			88,214	\$4,961.00
Boston, Mass.	20,708	2,632.00	30,000	\$675.00	954,716	38,664.00
Brazos de Santiago, Tex.						
Chicago, Ill.	6,606	1,011.00			95	1.00
Cincinnati, Ohio.	3,931	601.00				
Corpus Christi, Tex.						
Detroit, Mich.						
Duluth, Minn.	742	35.00				
Galveston, Tex.						
Key West, Fla.	30	6.20				
Middletown, Conn.						
Minnesota, Minn.						
New Orleans, La.						
New York, N. Y.	1,294,576	145,634.00	266,127	5,057.00	3,192,245	174,178.00
Oregon, Oreg.					420	4.00
Oswegatchie, N. Y.						
Paso del Norte, Tex.						
Philadelphia, Pa.	13,091	1,732.00			34,534	2,392.00
Saluria, Tex.						
San Francisco, Cal.						
Saint Louis, Mo.	8,101	1,232.00			986	51.00
Willamette, Oreg.					5,900	194.00
Total	1,298,883	154,383.20	296,127	5,732.00	4,277,110	220,415.00

FREE OF DUTY.

Customs districts and ports.	Mace.		Nutmegs.		Pepper, white and black.	
	Quantity.	Value.	Quantity.	Value.	Quantity.	Value.
	Pounds.		Pounds.		Pounds.	
Aroostook, Me						
Baltimore, Md.			22,314	\$8,634	429,849	\$57,075
Boston, Mass.	6,733	\$1,907.00	66,465	27,462	476,990	54,951
Brazos de Santiago, Tex.					460	102
Chicago, Ill.					23,746	3,575
Cincinnati, Ohio.					139,726	20,424
Corpus Christi, Tex.						
Detroit, Mich.						
Duluth, Minn.						
Galveston, Tex.						
Key West, Fla.	115	11.50				
Middletown, Conn.						
Minnesota, Minn.						
New Orleans, La.						
New York, N. Y.	115,022	28,617.00	1,008,282	395,583	9,119,301	1,309,084
Oregon, Oreg.						
Oswegatchie, N. Y.					1,465	338
Paso del Norte, Tex.						
Philadelphia, Pa.	139	118.00	177	162	195,455	25,702
Saluria, Tex.					25	8
San Francisco, Cal.			86,014	23,643	509,882	71,296
Saint Louis, Mo.			6,204	2,858	98,887	15,255
Willamette, Oreg.						
Total	122,009	30,653.50	1,189,450	458,342	10,995,786	1,557,810

SPICES AND CONDIMENTS.

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Statement showing, by customs districts and ports, the quantities and values of spices imported and entered for consumption, &c.—Continued.

FREE OF DUTY.

Customs districts and ports.	Pepper, cayenne.		Pimento.		All other, n. s. e., or p. f.	
	Quantity.	Value.	Quantity.	Value.	Quantity.	Value.
	Pounds.		Pounds.		Pounds.	
Aroostook, Me						
Baltimore, Md.						
Boston, Mass.			130	\$4	11,585	920.00
Brazos de Santiago, Tex.						
Chicago, Ill.						
Cincinnati, Ohio.						
Corpus Christi, Tex.	9,552	\$1,296			37,446	6,111.00
Detroit, Mich.						
Duluth, Minn.						
Galveston, Tex.						
Key West, Fla.					54	5.20
Middletown, Conn.						2,400.00
Minnesota, Minn.						
New Orleans, La.						
New York, N. Y.	723,459	73,789	2,500,523	109,605	33,600	
Oregon, Oreg.						
Oswegatchie, N. Y.						
Paso del Norte, Tex.	1,081	47				
Philadelphia, Pa.	79,118	5,503	130	6		
Saluria, Tex.						
San Francisco, Cal.					151,313	21,511.00
Saint Louis, Mo.					16,329	1,265.00
Willamette, Oreg.						
Total	813,210	80,635	2,500,783	109,615	250,327	32,212.20

DUTIABLE.

Customs districts and ports.	Mustard, ground or pre- served, in bottles, or o.		All other, ground or produced, n. s. e. or p. f.	
	Quantity.	Value.	Quantity.	Value.
	Pounds.		Pounds.	
Aroostook, Me	4½	\$1.35		
Baltimore, Md.	10,660	2,839.00		
Boston, Mass.	24,670	7,074.00	894	\$392.00
Brazos de Santiago, Tex.				
Chicago, Ill.				
Cincinnati, Ohio.				
Corpus Christi, Tex.				
Detroit, Mich.	896	278.00	6	1.00
Duluth, Minn.	5	1.00	20	5.00
Galveston, Tex.	1,800	317.00		349.41
Key West, Fla.	3	.42	2,762	246.00
Middletown, Conn.			3,312	9.00
Minnesota, Minn.			90	3,704.00
New Orleans, La.	45,207	8,810.00	36,982	498.00
New York, N. Y.	404,099	100,609.00	2,295	
Oregon, Oreg.				
Oswegatchie, N. Y.				
Paso del Norte, Tex.			442	70.62
Philadelphia, Pa.	106,735	30,701.00	420	157.00
Saluria, Tex.				
San Francisco, Cal.	38,985	11,195.00		
Saint Louis, Mo.	4,490	1,111.00		
Willamette, Oreg.			94½	5.26
Total	637,555½	162,936.77	47,317½	5,431.29

Unfortunately, with the exception of pepper and nutmeg, the tables giving the countries from which the imports were made do not distinguish between the spices. We are, however, able to see that a large portion of our supply comes through England, and the effect of this upon its quality is certainly not to improve it. The amount of each spice entered for consumption is visible in the last table and the port of arrival as well. New York of course receives the largest portion, followed by Boston and Baltimore, which are both milling centers.

As the spices are offered by the wholesale merchant they have a variable value, the quotations in New York for the week ending December 27, 1886, being as follows:

Kind of spices.	Price per pound.	Kind of spices.	Price per pound.
	<i>Cents.</i>		<i>Cents.</i>
Cassia:		Nutmegs, 110s.....	48 to 50
Batavia.....	7 to 7½	Pepper:	
China.....	5½	Singapore.....	18
Saigon:		West coast.....	15½ 16
Rolls.....	36 40	Acheen, prime.....	15
Broken.....	30	Penang, white.....	25 26
Chips.....	9 10	Singapore, white.....	28½ 29
Buds.....	10½ 10½	Red:	
Cloves:		Zanzibar.....	8½ 9
Prime.....	27	Bombay.....	6
Amboyna.....	27	Pimento, prime.....	5
Stems.....	6	Mustard:	
Ginger:		California:	
African.....	5	Brown.....	3½ 4½
Calcutta.....	3½ 4	Yellow.....	3½ 4½
Cochin.....	10 13	Dutch.....	5½ 6
Mace:		English.....	6¼ 6½
Batavia.....	50	Trieste, brown.....	5½ 5¾
Banda.....	50		
Penang.....	50		

It is of interest to examine these figures and compare them with the prices of wholesalers and retailers of ground spices.

The following figures show that prices alone are often a good indication of adulteration, the ground article being sold at wholesale or even retail for less than the cost of the pure unground spice.

Pure whole spices	Price per pound.	
	Wholesale.	Small lots.
	<i>Cents.</i>	<i>Cents.</i>
Ginger root:		
Bleached:		
Jamaica.....		16
American.....		14
Unbleached:		
Jamaica.....		14
Cochin.....	7½ to 11	12
Calcutta.....	3½ 4	8
Clove stems.....	6	10
Clove:		
Singapore.....	26½ 27	30
Amboyna.....	27	32
Pepper:		
White Singapore.....	24½	32
Best west coast.....	10½ 16	18
Acheen.....	15½	18
Mustard seed:		
Brown Trieste.....	5½ 5½	12
Yellow English.....	6½ 6½	12
California.....	3½ 4½	8
Brown California.....	3½ 4½	8
Cassia bark:		
Batavia.....	7 7½	10
Saigon stick.....	36 40	42
Saigon chip.....	9 10	10
Ligna sticks.....		9
Singapore black pepper.....	17½ 17½	
Zanzibar red pepper.....	9½	10.

Prices as supplied by spice mills.

Name.	Price per pound.	
	Wholesale, barrels.	Small lots in tin.
	<i>Cents.</i>	<i>Cents.</i>
Durham mustard.....	12	16
Extra strong mustard.....	16	20
Extra American mustard.....	18	22
B. Pure Pepper.....	12	16
Best pepper.....	10	14
Pure cinnamon.....	12	16
Best cinnamon.....	10	14
Pure ginger.....	7	11
Best ginger.....	6	10
Pure cayenne.....	12	16
Best cayenne.....	16	14
Pure allspice.....	6	10
Best allspice.....	5	9
Pure cloves.....	20	24
Best cloves.....	17	21
Pure mace.....	50	54
Pure white pepper.....	25	29

CHARACTER OF THE SPICES IN THE DISTRICT OF COLUMBIA.

The spices found in Washington are from various markets. The first class grocers carry the best English and rarely good American brands. Adulteration, however, is frequent, especially among the mustards, peppers, and cinnamon, the first having lost its oil and added flour, and the last having cassia substituted for it. Among the cheaper class of dealers adulterated spices are nearly universal, the supply being obtained largely from Baltimore and to a small extent ground in Washington, both places in which yellow corn and charcoal are much used as adulterants.

Of a series of samples collected impartially from all classes of shops the ratio of adulterated to non-adulterated was as follows:

Variety of spice.	Pure.	Adulterated.	Substituted.	Inferior or suspicious.
Cassia.....	3	1
Cinnamon.....	10
Cloves.....	2	9
Ginger.....	4	4
Mace.....	3	2
Mustard.....	10 *	1
Nutmeg.....	3
Pepper:
Black.....	1	9
White.....	2	2
Red.....	1	5
Pimento.....	5	3	1

*Oil expressed in one case and tumeric added and oil expressed in all American brands.

The preceding samples, which have proved to be so largely adulterated, have been used in connection with a collection of authenticated whole spices obtained directly from the importers as a means of investigating the methods which have been recommended for the detection

of adulterants, and the results of our examination of published methods and our own work are presented in the following pages, giving to the analyst a large amount of technical and scientific information which is of less interest to the general reader.

PART II.

THE DETECTION OF ADULTERATION OF SPICES AND CONDIMENTS.

In attempting to detect the adulteration of spices and condiments the methods which can be employed are of three kinds and depend upon the differences in structure between the adulterants and the substances to which they are added, and upon their proximate composition. The former differences are recognized by mechanical separation and the use of the microscope and the latter by chemical analysis. In the use of the microscope a knowledge of and ability to recognize the principal tissues which constitute the particular plant parts which are used as spices and also of those used as adulterants is necessary, while in the chemical examination the principles of proximate analysis must be understood and applied.

It is as necessary that the analyst should be thoroughly acquainted with the application of the microscope to the determination of cellular structure as to be able to make determinations of proximate principles in the substances under examination. In fact, a mechanical separation and microscopical examination is much more expeditious and more at the command of the majority of persons searching for adulteration.

Chemical analysis requires a systematic and extensive investigation of large numbers of samples, both pure and adulterated, to fix a standard of comparison, and this has hitherto been seldom done, owing to the elaborate nature of the work and the expense involved. It should not be neglected, however, since it serves as a most certain confirmation of the microscopic results, besides furnishing information in regard to the quality of the specimen examined and as to the quantity of any adulterant, which cannot be obtained in any other way. While, therefore, the microscopic method will always retain its value for preliminary and qualitative examinations, it must, with the development of the means of chemical investigation, become more and more a mere adjunct of the latter, as in fact the microscope has become in all branches of science. The application of the microscope to the detection of adulterants will, therefore, be considered first.

MECHANICAL SEPARATION AND MICROSCOPIC EXAMINATION OF SPICES AND CONDIMENTS.

As a preliminary to the microscopic examination a mechanical separation by means of sieves of different mesh furnishes a means of detecting adulterants and selecting particles for further investigation,

which is of the greatest value and often reveals without additional means the presence of foreign materials. Many adulterants are not ground as fine as the spice to which they are added, and by passing the substance through a sieve of from 40 to 60 meshes to the inch the coarser particles remaining will either be recognized at once by the unaided eye or with a pocket lens or the microscope. In this way turmeric is readily separated from mustard, and yellow corn, mustard hulls and cayenne from low-grade peppers; in no case was the aid of more than an ordinary pocket lens necessary for subsequent recognition, although higher powers of the microscope were confirmatory.

Without entering here into the practical manipulation of the microscope, it may be said that for the purposes of the food analyst it is only necessary to have a stand of good workmanship, not necessarily, though preferably, furnished with substage condenser, but supplied with Nicol prisms for the use of polarized light. Objectives of inch, half-inch, or, for some of the starches, one-fifth inch equivalent focus are sufficient. One eye-piece of medium depth is also enough. It is also desirable to be provided with a dissecting microscope for selecting particles for examination from large masses of ground spice. For those who can afford it, the large stand of Zeiss made for this purpose proves most useful, but simpler forms or even a hand lens will serve perfectly well.

For smaller apparatus it is unnecessary to provide anything aside from what is found in ordinary laboratories. A few beakers, watch glasses, stirring rods, and specimen tubes, with bottles for reagents will be sufficient in addition to the ordinary glass slides and cover glasses.

The reagents which are required include, in case no permanent mounts are required:

Alcohol, strong.

Ammonia.

Chloralhydrat, solution 8 parts to 5 of water.

Glycerine.

Iodine solution: water 15 parts, iodide of potash 20 parts, iodine 5 parts.

Water, distilled.

Schulze's reagent, a mixture of chlorate of potash and dilute nitric acid prepared as wanted.

Balsam in benzol and glycerine jelly are desirable for mounting media and some sheet wax for making cells.

In addition the analyst should supply himself with specimens of whole spices, starches, and known adulterants which can be used to become acquainted with the forms and appearances to be expected. It is easier to begin one's study in this way on sections prepared with the knife than upon the powdered substance, and it is often necessary to refer to them for comparison in the examination of trade samples.

PHYSIOLOGICAL STRUCTURE IN THE SPICES AND THEIR ADULTERANTS.

The vegetable tissues which made up the structure of the spices, and the material of a vegetable origin which are added as adulterants, consist of cells of different forms and thickness. Those which are most

prominent and common are the parenchyma, the sclerenchyma, fibrous tissue, and the fibro-vascular bundles. Spiral and dotted vessels are also common in several of the adulterants, and in the epidermis other forms of tissue which it is necessary to be well acquainted with though not physiologically. The parenchyma is the most abundant tissue in all material of vegetable origin, making up the largest proportion of the main part of the plant. It is composed of thin-walled cells, which may be recognized in the potato and in the interior of the stems of maize. In the latter plant, also, the fibro-vascular system is well exemplified, running as scattered bundles between the nodes or joints, and easily made out.

Fibrous tissue consists of elongated thick-walled cells or fibers which are very common in the vegetable kingdom and are well illustrated in flax. They are not as common in spices as in the adulterants. They are optically active, and in the shorter forms somewhat resemble the cells next described. They are seen in one of the coats of buckwheat hulls and in the outer husk of the cocoanut.

The sclerenchyma is found in the shells of many nuts and in one or two of the spices. The cells are known as stone cells, from the great thickening of their walls, and to them is due the hardness of the shell of the cocoanut, the pits of the olive, &c. Their structure is illustrated in Fig. 5 from Strasburger.

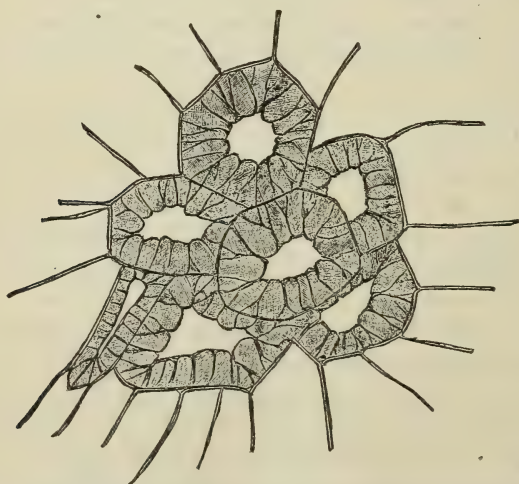


FIG. 5. Sclerenchyma or stone cells. $\times 240$. (After Strasburger.)

Spiral and dotted vessels are common in woody tissue and are readily recognized. With all these forms the analyst should familiarize himself, and as an aid may consult Bessey's Botany in the American Science Series.

In pepper and in mustard the parenchyma cells are prominent in the interior portion of the berry, while those constituting the outer coats are indistinct from their deep color in the pepper, but in the mustard characteristic of the particular species. In fact, in many of the spices, especially those which are seeds, the forms of the epidermal cells are very striking, and even if no attempt is made to classify them their peculiarities must be carefully noted, as the recognition of the presence of foreign husky matter depends upon a knowledge of the normal appearance in any spice. The fibro-vascular bundles are most prominent in ginger and in the barks, where in the powdered spices they are found as stringy particles.

The sclerenchyma or stone cells, are commoner in the adulterants, especially in cocoanut shells, where may also be seen numerous spiral cells and in the exterior coats fibrous tissue.

As aids to distinguishing these structures, the following peculiarities may be cited.

The stone cells and fibrous tissue are optically active and are therefore readily detected with polarized light, shining out in the dark field of the microscope as silver white or yellowish bodies.

The fibro-vascular bundles are stained deep orange brown with iodine, owing to the nitrogenous matter which they contain, while parenchyma is not affected by this reagent aside from the cell contents, nor has it any action on polarized light, remaining quite invisible in the field with crossed prisms.

STARCH.

Aside from the cellular tissue, starch is the most important element in the plant for the analyst, and its peculiarities will be considered quite fully.

It possesses an organized structure and is distinguished by its reaction with iodine solution, with which it strikes a deep blue or blackish blue color, varying somewhat with different kinds of starch and with the strength of the reagent.

Conversely its absence is marked by no blue color under the same circumstances. Heat, however, as in the process of baking, so alters starches, converting them into dextrine and related bodies, that they give a brown color with iodine instead of a blue black. They are then in fact no longer starch, although their form, often not being essentially changed, permits of their identification.

Although a practical experience in recognizing the starches by these characteristics is essential for their rapid detection when occurring as adulterants, a valuable guide may be supplied to a certain extent by artificial classifications, such as Vogel, Muter, and Blyth, after Tripe's work, have arranged.

Vogel's and Muter's are based on the form and size of the granules, of the hilum or central depression or nucleus and the prominence and position of the rings. Tripe showed that with polarized light and

selenite the starches of tubers showed a more varied play of colors than the cereal and leguminous starches which are produced above ground. On this fact Blyth has made another classification. Both are of value and interest.

Vogel's table of the different starches and arrowroots of commerce.

A.—Granules simple, bounded by rounded surfaces.

- I. Nucleus central, layers concentric.
 - a. Mostly round or from the side lens-shaped.
 1. Large granules .0396 - .0528^{mm}, *rye starch*.
 2. Large granules .0352 - .0396^{mm}, *wheat starch*.
 3. Large granules .0264, *barley starch*.
 - b. Egg-shaped, oval, kidney-shaped. Hilum often long and ragged.
 1. Large granules .032 - .079^{mm}, *leguminous starches*.
- II. Nucleus eccentric, layers plainly eccentric or meniscus shaped.
 - a. Granules not at all or only slightly flattened.
 1. Nucleus mostly at the smaller end .06 - .10^{mm}, *potato starch*.
 2. Nucleus mostly at the broader end or towards the middle in simple granules .022 - .060^{mm}, *maranta starch*.
 - b. Granules more or less strongly flattened.
 1. Many drawn out to a short point at one end.
 - a. At most .060^{mm} long, *curcuma starch*.
 - b. As much as .132^{mm} long, *canna starch*.
 2. Many lengthened to bean-shaped, disk-shaped, or flattened; nucleus near the broader end .044 - .075^{mm}, *banana starch*.
 3. Many strongly kidney-shaped; nucleus near the edge .048 - .056^{mm}, *sisyrinchium starch*.
 4. Egg-shaped; at one end reduced to a wedge, at the other enlarged; nucleus at smaller end .05 - .07^{mm}, *yam starch*.

B.—Granules simple or compound, single granules or parts of granules, either bounded entirely by plane surfaces, many angled, or by partly round surfaces.

- I. Granules entirely angular.
 1. Many with prominent nucleus. At most .0066^{mm}, *rice starch*.
 2. Without a nucleus. The largest .0038^{mm}, *millet starch*.
- II. Among the many angled also rounded forms.
 - a. No partly rounded forms present, angular form predominating.
 1. Without nucleus or depression very small, .0044^{mm}, *oat starch*.
 2. With nucleus or depression .0132 - .0220^{mm}.
 - a. Nucleus or its depression considerably rounded; here and there the granules united into differently formed groups, *buckwheat starch*.
 - b. Nucleus mostly radiatory or star-shaped; all the granules free, *maize (corn) starch*.
 - b. More or less numerous kettle-drum and sugar-loaf like forms.
 1. Very numerous eccentric layers; the largest granules .022 - .0352^{mm}, *batata starch*.
 2. Without layers or rings .08 - .022^{mm}.
 - a. In the kettle drum-shaped granules the nucleal depression mostly widened on the flattened side, .008 - .022^{mm}, *cassava starch*.
 - b. Depression wanting or not enlarged.
 - aa. Nucleus small, eccentric, .008 - .016^{mm}, *pachyrhizus starch*.
 - bb. Nucleus small, central, or wanting.
 - aaa. Many irregular angular forms .008 - .0176^{mm}, *sechium starch*.
 - bbb. But few angular forms; some with radiatory nucleal fissure, .008 - .0176, *castanospermum starch*.

C.—Granules simple and compounds, predominant forms, egg form and oval, with eccentric nucleus and numerous layers, the compound granule made up of a large granule and one or more relatively small kettledrum-shaped ones, .025 - .066^{mm}, *sago starch*.

*Muter's table for the detection of starches when magnified about 230 diameters.**

[All measurements are given in decimals of an inch.]

Group I: All more or less oval in shape and having both hilum and rings visible.

Name.	Shape.	Normal measurements.	Remarks.
Tous les mois.....	Oval, with flat ends.....	.00370 to .00185	Hilum annular, near one end and incomplete rings.
Potato.....	Oval.....	.00270 .00148	Hilum annular, rings incomplete, shape and size very variable.
Bermuda arrowroot.....	Sack-shaped.....	.00148 .00129	Hilum distinct annular, shape variable, rings faint.
St. Vincent arrowroot.....	Oval-oblong.....	.00148 .00129	Hilum semilunar, rings faint, shape not very variable.
Natal arrowroot.....	Broadly ovate.....	.00148 .00129	Hilum annular, in center and well marked complete rings.
Galangal.....	Skittle-shaped.....	†.00135	Hilum elongated, very faint incomplete rings.
Calumba.....	Broadly pear-shaped....	†.00185	Hilum semilunar, faint but complete rings, shape variable.
Orris root.....	Elongated-oblong.....	†.00092	Hilum faint, shape characteristic.
Turmeric.....	Oval-oblong, conical....	†.00148	Very strongly-marked incomplete rings.
Ginger.....	Shortly conical, with rounded angles.	†.00148	Hilum and rings scarcely visible, shape variable but characteristic.

Group II: With strongly-developed hilum more or less stellate.

Bean.....	Oval-oblong.....	†.00135	Fairly uniform.
Pea.....	Like bean.....	.00111 — .00074	Very variable in size, with granules under .00111 preponderating.
Lentil.....	Like bean.....	†.00111	Hilum, a long depression seldom radiate.
Nutmeg.....	Rounded.....	†.00055	The small size and rounded form distinctive.
Dari.....	Elongated hexagon.....	†.00074	Irregular appearance and great convexity distinctive.
Maize.....	Round and polygonal....	†.00074	The rounded angles of the polygonal granules distinctive.

Group III: Hilum and rings practically invisible.

Wheat.....	Circular and flat.....	.00185 to .00009	Very variable in size and very dull polarization in water.
Barley.....	Slightly angular circles	†.00073	The majority measuring about .00073 distinctive.
Rye.....	Like barley.....	.00148 .00009	Small granules, quite round, and here and there cracked.
Jalap.....	Like wheat.....	Polarizes brightly in water.
Rhubarb.....do.....	.00055 §.00033	Polarizes between jalap and wheat, and runs smaller and more convex.
Senega.....do.....	.00148 — .00009	Measurements the only guide.
Bay berry.....do.....	.00074 — .00011	
Sumbul.....do.....	.00074 — .00009	

*Analyst 1, 172-174, November 15, 1876.

†About.

‡And a few four times this size.

§ For small granules.

Muter's table for the detection of starches, &c.—Continued.

Group III: Hilum and rings practically invisible—Continued.

Name.	Shape.	Normal measurement.	Remarks.
Chestnut	Very variable.....	.00090 — .00009	Variable form, and small but regular size, distinctive.
Acorn	Round-oval	*.00074	Small and uniform size, distinctive.
Calabar bean	Oval-oblong00296 .00180	Large size and shape characteristic.
Liquorice.....	Elongated-oval	*.00018	Small size and shape distinctive.
Hellebore (green or black)...	Perfectly rotund.....	.00037 .00009	Small, regular size and roundity, distinctive.
Hellebore (white)	Irregular.....	.00055 .00009	Irregular shape and faint central depression, distinctive.

Group IV: More or less truncated at one end.

Cassia	Round.....	.00111 to .00018	Round or muller shaped granules and faint circular hilum.
Cinnamon	Like cassia00074 .00009	More frequently truncated than cassia, and smaller.
Sago (raw).....	Oval-ovate00260 .00111	Has circular hilum at convex end and rings faintly visible.
Sago (prepared)do00260 .00111	Has a large oval or circular depression, covering one-third nearly of each granule.
Tapioca	Roundish.....	.00074 .00055	A little over 50 per cent. truncated by <i>one</i> facet, and a pearly <i>hilum</i> .
Arum.....	Like tapioca	*.00056	Smaller than tapioca and truncated by <i>two</i> facets.
Belladonnado		Not distinguishable from tapioca.
Colchicum.....do	*.00074	Larger than tapioca, and contains many more truncated granules.
Scammony.....do	*.00045	Smaller than tapioca, more irregular, and <i>hilum</i> not visible.
Canella.....	Very variable00033 — .00022	Very variable, form and small size the only points.
Podophyllum	Like tapioca	*.00040	Like scammony, but has visible <i>hilum</i> in most of the granules.
Aconitedo	*.00037	Like tapioca, but half the size.

Group V: All granules more or less polygonal.

Tacca	Poly or hexagonal.....	.00075 to .00037	Distinguished from maize by its <i>sharp</i> angles.
Oat	Polygonal	*.00037	Larger than rice and hilum visible in some granules.
Ricedo00030 — .00020	Measurement using one-eighth or one-twelfth inch-power, and then hilum visible.
Pepperdo00020 — .00002	Do.
Ipecacuanha.....do	*.00018	Some round and truncated granules, adhering in groups of three.

*About.

Blyth's classification.

DIVISION I.—Starches showing a play of colors with polarized light and selenite plate.

Class I. The hilum and concentric rings clearly visible, all the starches oval or ovate, including tous les mois, potato, arrowroot, calumba, orrisroot, ginger, galangal, and turmeric.

DIVISION II.—Starches showing no iridescence, or scarcely any, when examined by polarized light and selenite.

Class II. The concentric rings all but invisible, hilum stellate, including bean, pea, maize, lentil, dari, and nutmeg.

Class III. Starches having both the concentric rings and hilum invisible in the majority of granules. This important class includes wheat, barley, rye, chestnut, acorn, and many starches in medicinal plants.

Class IV. All the granules truncated at one end. This class includes sago, tapioca and arum, several drugs, and cinnamon and cassia.

Class V. In this class all the granules are angular in form and it includes oats, tacca, rice, pepper, as well as ipecacuanha starch.

Of the starches which are included in the preceding classification but a limited number will be met with in spices and their adulterants or in the commoner foods. One must, however, be able to readily recognize the following :

Starches natural to spices and condiments.	Starches of admixture.
Ginger. Pepper. Nutmeg. Cassia. Pimento. Cinnamon. Cayenne.	Wheat and other cereals. Corn. Oats. Barley. Potato. Maranta and other arrowroots. Rice. Bean. Pea. Sago. Buckwheat.

The remainder may be found in other foods and in drugs and cannot well be omitted therefore from our classifications.

No one of these is complete in itself, but from the characters given and with the aid of our illustrations the starches which commonly occur in the substances which are here considered may usually be identified without difficulty.

In practice the manipulation of the microscope and the preparation of the object requires some little experience, but not more than analysts usually have had. For the benefit of those who have had none, it may be said that a small portion of the starch or spice is taken up upon a clean camel's hair brush and dusted upon a common slide. The excess is blown away and what remains moistened with a drop of a mixture of equal parts of glycerine and water or glycerine and camphor-water and covered with a cover glass. It is well to have a small supply of the common starches in a series of tubes, which can be mounted at any moment and used for comparison. They can be permanently mounted by making with cork borers of two sizes a wax cell-ring equal to the diameter of the cover glass, and after cementing the cell to the slide with

copal varnish thinned with turpentine and introducing the starch and glycerine mixture, fixing the cover glass on after running some of the cement over the top of the ring. A little experience will enable one to put the right amount of liquid in the cell and to make a preparation which will keep for some time. After several months, however, it is difficult to distinguish the rings which mark the development of the granule, and although for reference as to size and form the preparation is satisfactory, it is always advisable in doubtful cases to examine some fresh material.

For other purposes the starches should be mounted in prepared Canada balsam or dammar by well-known methods. In this medium they can be preserved indefinitely, but are scarcely visible with ordinary illumination, and must be viewed by polarized light, which brings out distinctive characters, not seen as well or at all in other mounts.

Appearance in glycerine and water.—When mounted in the manner already described, or in water alone if for only temporary use, and examined under a microscope with an objective of equivalent focus of one-half to one-fifth inch, and with means for oblique illumination, the starches will display the characteristics which have been mentioned, and which are illustrated on Plates 26, 27, and 28. These illustrations have been drawn from nature by Dr. George Marx, and represent the starches as nearly as possible, as they are seen, and not as in many of the absurd illustrations of the handbooks of microscopists of the past and present day, which are entirely ideal, representing the granules not as extremely translucent bodies, but with the rings or layers as strongly-marked lines. Examined in this manner the size, shape, presence or absence of a nucleus or hilum, and of the rings and their arrangement, can be made out, and the starch referred to its proper position.

Appearance in balsam with polarized light.—Mounted in balsam the starches are scarcely visible under any form of illumination with ordinary light, the index of refraction of the granules and the balsam being so nearly alike. When, however, polarized light is used the effect is a striking one, and is illustrated in Plates 19 to 21. It is very easy to distinguish all the characteristics, except the rings, the center of the cross being at the nucleus of the granule.

With the selenite plate a play of colors is produced, which is peculiar to some of the starches and forms the basis of Blyth's classification.

The principal starches which are met with may be described as follows, in connection with our illustrations, beginning with those of the arrowroot class, including the potato, ginger, and turmeric.

Potato starch.—The starch grains of the potato are very variable in size, being found from .05 to .10^{mm} in length, and in shape from oval and allied forms to irregular and even round in the smallest. These variations are illustrated in Fig. 57, but the frequency of the smaller granules is not as evident as in Figs. 30 and 31. The layers are visible in some granules with great distinctness and in others hardly at all, being rather

more prominent in the starch as obtained from a freshly cut surface. The rings are more distinct, too, near the hilum or nucleus, which in this, as in all tuberous starches, is eccentric, shading off toward the broader or more expanded portion of the granule. The hilum appears as a shadowy depression (Fig. 57) and with polarized light its position is well marked by the junction of the arms of the cross, and it will be found by comparison of Figs. 31 and 32, on Plate XVI, that in the potato it is oftener at the smaller end of the granule and in the arrowroot at the larger. With polarized light and a selenite plate the beautiful play of colors is obtained which is the basis of Blyth's classification. The smaller granules, which are nearly round, may readily be confused with other starches, but their presence serves at once to distinguish this from *Maranta* or *Bermuda arrowroot* starch.

Rarely compound granules are found composed of two or three single ones each with its own nucleus.

Of the same type as the potato starch are the various arrowroots, the only one of which commonly met with in this country being the Bermuda, the starch of the rhizome of *Maranta arundinacea*, and the starch of Turmeric.

Maranta starch.—The granules are usually not so varied in size or shape as those of the potato, as may be seen in Figs. 30, 31, and 32, averaging about .07^{mm} in length. They are about the same size as the average of the latter, but are never found as large or as small, which, together with the fact that the end at which the nucleus appears is broader in the *Maranta* and more pointed in the potato, enables one to distinguish the starches without difficulty. With polarized light the results are similar to those seen with potato starch, and this is a ready means of distinguishing the two varieties, by displaying in a striking way the form of the granule and position of the hilum, as is illustrated in Figs. 31 and 32.

Curcuma or turmeric starch.—Turmeric contains a starch (Fig. 63, Plate XXVIII), which, although of the arrowroot class, is quite distinct in appearance from those which we have described. It is most irregular in outline, so that it is impossible to define its shape or to do more than refer to the illustration. Many of the granules are long and narrow and drawn out to quite a point. The rings are distinct in the larger ones. The size is about that of the *Maranta*.

Ginger starch (Figs. 41 and 42, Plate XXI, and Fig. 58, Plate XXVII).—This starch is of the same class as those from the potato and *Maranta* and several others which are of under-ground origin. In outline it is not oval like those named, but more rectangular, having more obtuse angles in the larger granules and being cylindrical or circular in outline in the smaller.

It averages nearly the same size as *Maranta* starch, but is much more variable both in size and form. The rings are scarcely visible even with the most favorable illumination.

Sago starch.—This exists in two modifications in the market; as raw and as prepared sago. In the prepared condition it is characterized by a larger circular depression in the center of most of the granules. The rings are not visible. They are mostly circular in form or approaching it, and vary from .025 to .065^{mm} in diameter.

Leguminous starches, pea and bean (Figs. 39 and 40, Plate XX, and Figs. 60 and 61, Plate XXVII).—These starches produce but a slight effect with polarized light. The rings are scarcely visible, and the hilum is stellate or much cracked along a median line; the bean more so than the pea, the latter resembling fresh dough kneaded again into the center as in making rolls, and the former the shape assumed by the same after baking. They are both somewhat variable in size, ranging from .025 to .10^{mm} in length.

Nutmeg starch (Fig. 64, Plate XXVIII).—This starch, which in some respects resembles the preceding—the rings being scarcely visible and not iridescent with polarized light—is much smaller in size and quite variable. The larger granules are at times as long as .05^{mm} and the smallest smaller than .005^{mm}. They are of extremely irregular form, with angular depressions and angular outlines, and are distinguished by a budded appearance, caused by the adherence of small granules to the larger.

Capsicum starch (Fig. 67, Plate XXVIII) is nearly circular or rounded, polyhedral in form, with scarcely visible rings, and in most cases a depressed hilum resembling in size and shape corn starch, but having peculiar irregularities, which distinguish it, such as a rosette-like formation on a flattened granule or a round depression at one end. It does not polarize as actively as maize starch, and can be distinguished from rice by the greater angularity of the latter.

Pepper starch (Fig. 65, Plate XXVIII) is the most minute starch that is usually met with, not averaging over .001^{mm}, nor exceeding .005. It is irregularly polyhedral, polarizes well, but requires a high power to discover any detail when a hilum is found. It cannot be confused with other starches.

Cinnamon starch (Fig. 46, Plate XXIV, and Fig. 66, Plate XXVIII) has an extremely irregular, polyhedral or distorted granule, often united in groups with smaller granules adherent to the larger ones. In size it varies from .001 to .025, averaging nearly the latter size. In some granules a hilum can be distinguished, but no rings. It is readily detected with polarized light.

Buckwheat starch (Fig. 62, Plate XXVIII) is very characteristic. It consists of chains or groups of angular granules, with a not very evident circular nucleus and without rings. The outline is strikingly angular and the size not very variable, being about .01 to .015.

Maize or corn starch (Fig. 33, Plate XVII, and Fig. 54, Plate XXVI).—The granules of corn-starch are largely of the same size, from .02 to .03^{mm} in diameter, with now and then a few which are much smaller. They are mostly circular in shape or rather polyhedral, with rounded

angles. They form very brilliant objects with polarized light, but with ordinary illumination show but the faintest sign of rings, and a well-developed hilum, at times star-shaped, and at others more like a circular depression.

Rice starch (Figs. 35 and 36, Plate XVIII, and Fig. 55, Plate XXVI) is very similar to corn starch, and easily confused with it, being about the same size. It is, however, distinguished from it by its polygonal form, and its well-defined angles. The hilum is more prominent and more often stellate or linear. Several granules are at times united.

Wheat starch (Fig. 34, Plate XVII, and Fig. 50, Plate XXVI) is quite variable in size, varying from .05 to .012^{mm} in diameter. It belongs to the same class as barley and rye, the hilum being invisible and the rings not prominent. The granules are circular disks in form, and there are now and then contorted depressions resembling those in pea starch. It is the least regular of the three starches named and does not polarize actively.

Barley starch (Fig. 37, Plate XIX, and Fig. 51, Plate XXVI) is quite similar to that of wheat, but does not vary so much in size, averaging .05^{mm}; has rings which are much more distinct, and very small granules adhering to the largest in bud-like forms.

Rye starch (Fig. 52, Plate XXVI) is more variable in size, many of the granules not exceeding .02^{mm}, while the largest reach .06 to .07^{mm}. It lacks distinctive characteristics entirely, and is the most simple in form of all the starches we have described.

Oat starch (Fig. 38, Plate XIX, and Fig. 53, Plate XXVI) is unique, being composed of large compound masses of polyhedral granules from .12 to .02^{mm} in length, the single granules averaging .02 to .015 mm. It does not polarize actively as may be seen in figure and plate, and displays neither rings nor hilum. The illustration shows its nature with accuracy.

Our descriptions, it will be seen, do not agree entirely with those of other authors, which in the same way do not agree among themselves.

This shows a variation in the peculiarities of size, shape, &c., which must be carefully allowed for, and the necessity for every investigator to compare a starch which he is desirous of identifying with authentic specimens.

STRUCTURE AND PECULIARITIES OF THE COMMONER ADULTERANTS.

Before proceeding to the consideration of the normal structure and composition of the spices and condiments and the adulterations detected in commercial specimens, it is well to become familiar with the characteristics of the common adulterants and materials which are liable to be used for this purpose.

The starches have been already described and their value as a means of identifying different vegetable materials noted. By this means we are able to detect the different cereals which are often added as diluents.

Maize, or corn as it is commonly known, is a common adulterant. By selecting particles from the ground material and crushing them the character of the starch may be recognized, but in a cursory examination the first sign of the presence of this cereal is the discovery of one of the thin outer coats of the grain which becomes detached in milling and, being tough, is not readily reduced. In yellow corn it has a peculiar pinkish color and simple structure of longitudinal cells. One should learn to recognize it from a specimen ground for the purpose.

Rice, which in its broken unmarketable form is sometimes used as a diluent, may be recognized by the brilliant appearance of the hard white particles which must be picked out of the spice under a hand lens, crushed, and examined as usual. Rice bran has not been met with.

The two cereals named are the only ones which are commonly met with which introduce starch. Rarely clean *wheat bran* is added, which can be recognized by its distinctive structural character, illustrated in many hand-books, but which can be learned much better from an authentic specimen, which should be soaked in chloral hydrate.

As modified cereals, we find refuse bread, cracker-dust, and ship-bread in which the wheat starch is much changed from its original form by the heat and moisture of the cooking process so that at times it might be confused with a leguminous starch. The softness of the particles and the ease with which they fall to pieces in water reveals the nature of the material. It is a common diluent.

Oil-seed, oil-cake, and husks are very commonly used in many parts of the country for purposes of sophistication. They are most readily recognized by the peculiar structure of the outer coats of the seed. The particles which can usually be found and selected with a dissecting microscope should be examined in alcohol or glycerine, or a mixture of the two, as the outer coats of some seeds, such as mustard, are swollen by water and become indistinct. The appearance of mustard hulls is given on page 172, and the many varieties of the cruciferous seeds resemble it much, so that it is difficult to distinguish them, which is, however, not important. They are generally distinguished by the outer layer of hexagonal cells, and a middle and an inner coating which consist of peculiar angular cells, the latter much larger than the former, which are the most characteristic, and should be compared with specimens of seed of known origin. The structure of some of them is diagrammatically presented in fig. 6, from Schimper. After soaking in chloral hydrate the remaining interior layers are perhaps more easily made out, and in some cases after moderate bleaching with nitric acid and chlorate. The interior of these seeds is not blued by iodine.

Peanut or groundnut cake is recognized by the characteristic structure of the red-brown coat which surrounds the seed, which consists of polygonal cells with peculiar saw-toothed thickening of the walls. The seed itself consists of polygonal cells, full of oil and starch granules, which are globular in form and not easily confused with pepper starch.

The structure of the brown membrane is best made out in chloral hydrate, which removes the red color and leaves the fragments of a bright yellow.

Linseed cake distinguished by the fact that its husk is made up of one or two characteristic elements. The outer coat or epidermis is colorless and swells up in water, forming a mucilage like the mustard seed. Beneath this is a layer of thin round yellow cells, while the third is very characteristic, and consists of narrow, very thick-walled dotted vessels. Next to these is an inner layer of compact polygonal cells, with fairly thin but still thickly dotted white walls and dark-brown contents, containing tannin. The endosperm and embryo are free from starch; nor are they colored yellow by potash, as is the case with mustard and rape cake.

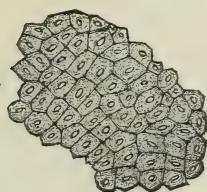


FIG. 6.

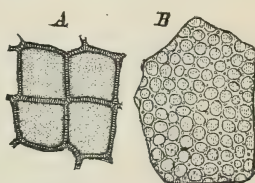


FIG. 7.

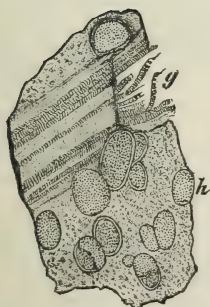


FIG. 8.

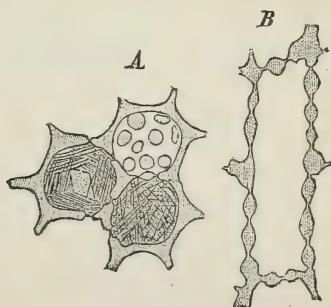


FIG. 9.

FIG. 6. Rape-seed husk (exterior surface).

FIG. 7. Linseed husk. A more, B less magnified.

FIG. 8. Almond shell fragment. h hairs, g spiral vessels. $\times 70$.

FIG. 9. Palm-seed. Interior. $\times 240$. (After Schimper.)

Palm-cake is probably not common enough in this country to be used as an adulterant; nor are *olive stones*, which as they consist, after bleaching with Schulze's reagent, almost entirely of very thick stone cells, are easily made out under polarized light.

Cocoanut shells are often used, it seems from the evidence of the Canadian analysts. They are similar to the olive stones in structure but more complicated, as in addition to the numerous short stone cells there are many long ones with thinner walls, and here and there spiral vessels, from the fibrous tissue, all of which are only readily seen after

bleaching. When the shells are roasted or charred they refuse to bleach, and it is then only possible to class the particles on which the reagents do not act as roasted shells or charcoal. They are frequently used in peppers to give color to material rendered too light by white adulterants.

The composition of these substances is shown in the following determination which reveal the effect of their addition upon the normal composition of the spices:

	Olive stones.	Cocoanut shells.
Water	5.63	6.15
Ash	4.28	2.13
Fiber	41.33	37.15
Albuminoids	1.56	1.25
Nitrogen25	.20

More in regard to olive stones will be learned when the discussion of the adulteration of pepper is considered.

Buckwheat hulls after bleaching with Schulze's reagent show a preponderance of tissue made up of long, slender, and pointed sclerenchyma cells and a smaller amount of reticulated tissue resembling the cereals somewhat and cayenne pepper. Portions of the endosperm or interior of the seed are also visible, and consist of an agglomeration of small hexagonal cells which originally contained starch. The starch is readily recognized by its peculiar characteristics. The sclerenchyma is, of course, optically active and forms a beautiful and distinctive object with polarized light.

Sawdust of various woods may be recognized by the fragments of various spiral and dotted vessels and fibrous material which are not found in the spices or other adulterants.

Bark, in some parts of the world a common addition to pepper, is detected by its stone cells, which are larger than those of pepper, and of different form and more numerous, and by its fibrous vessels which are made out readily after bleaching. The source of a particular bark cannot, however, be made out.

Rice bran.—This, as several other similar products, is made up prominently of two series of cells at right angles to each other, which make up the outer coats of the grain. The structure is best made out after soaking in chloral-hydrate. The cells of one series are long, small, and thin-walled, and are arranged in parallel bundles. The others have very much thickened walls, and are only two or three times as long as broad. They are at times distinguished, for convenience, as the longitudinal and transverse cells. The remaining layers of the bran are not prominent.

Clove stems, used frequently as a diluent, can be distinguished by their peculiar yellow-dotted vessels and their large and quite numerous stone-

cells, neither of which are seen prominently in the substances which are adulterated.

These suggestions of the peculiarities of the different adulterants should, of course, be confirmed and supplemented, and the eye accustomed to recognize their structure by means of a study of the actual substances, which should always be at hand in the dry and ground condition for reference. Quickness and certainty will be much advanced by such facilities.

Reference may also be fittingly made here to chemical operations of a general nature, which are applicable to all the spices.

CHEMICAL EXAMINATION.

Determinations of a quantitative nature should include—

Water.—A portion of the powdered spice which should pass a 60-mesh sieve, one gram, is to be dried at 105° to 110° C. in an air bath, provided with a regulator, until on successive weighings a gain is found showing that oxidation has begun. Twelve hours, or overnight, usually suffices. The loss is water, together with the largest part of the volatile oil. Deduction of the volatile oil, as determined in the ether extract, will give a close approximation to the water.

Ash.—In the same portion the ash is determined by incineration at a very low temperature, such as may be best obtained in a gas muffle, which is a most convenient arrangement for work of this description, and far superior to any kind of lamp or burner. The proportion of ash insoluble in acid may also be determined where there is reason to believe that *sand is present*.

Volatile oil and ether extract.—Two grams of the substance are extracted for twenty hours in a siphoning extraction apparatus on the Soxhlet principle, with Squibbs's best ether. The apparatus we use in this laboratory, arranged by Mr. A. E. Knorr, he describes as follows:

The substance under examination is placed in a test tube, which is then inserted into a continuous extraction apparatus of the intermittent siphon class. The tubes used for this purpose are ordinary test tubes, the bottom of which has been blown out. A wad of washed cotton of sufficient thickness is put into the lower end of the tubes to prevent any solid particles of the sample from finding their way into the receiving flask; another wad of cotton is packed on top of the sample, and the apparatus is then so adjusted that the condensed ether drops into the tube, and, percolating through the sample, siphons into the receiving flask, when the operation is continued *ad infinitum*.

It is absolutely necessary to use the best Squibbs's ether in order to avoid extracting substances other than oil and soluble in alcohol, and to continue the extraction for at least the time named, as piperine and several other proximate principles are not extremely soluble in ether. If these precautions are followed we have found no difficulty in extracting all the piperine, for example, and obtaining duplicate results of

great accuracy. On stopping the extraction the extract is washed into a light weighed glass dish, and the ether allowed to evaporate spontaneously and not too rapidly, as in the latter case water is condensed in the dish, which it is difficult to remove. When the ether has disappeared—which ought not to take too long, as in that case some oil is volatilized—the dish is placed in a large dessicator, with pumice and sulphuric acid—chloride of calcium having been shown to be useless*—and allowed to remain overnight, to remove any moisture. The loss of oil by this process is hardly appreciable. The dish is then weighed, and afterwards heated to 110° C. for some hours, to drive off the volatile oil, beginning at a rather low temperature, as the oil is easily oxidized, and then is not volatile. The residue is weighed, the difference being calculated to volatile oil, and then examined as to its composition of purity. The results are fairly satisfactory, as appears from the following duplicates:

Duplicate fat and volatile oil determinations in spices.

No.	Percent.	Percent.	No.	Per cent.	Per cent.
4629.....	5.49	8.38	4632.....	4.11	3.66
4629.....	5.23	8.51	4632.....	3.93	4.22
4630.....	5.15	6.54	4633.....	5.06	6.94
4630.....	4.72	6.50	4633.....	5.33	7.68
4631.....	6.64	13.19	4634.....	5.15	3.50
4631.....	6.84	12.14	4634.....	2.26	3.59

Alcohol extract.—This may be made in the same manner as the ether extract, using, of course, the substance already extracted. The solvent may be either absolute alcohol, that of 95 per cent. by volume, or 80 per cent. by weight. The latter is preferable in most cases, as there is no definite point with the stronger spirit at which the extraction is complete.

In the investigation of spices merely for adulteration this extraction is of little value.

Starch, &c., reducing sugars.—The amount of reducing material produced by boiling the spices with dilute acid serves with several as an index of purity. In the case of pepper, which contains naturally a large amount of starch, the addition of the common fibrous adulterants reduces the equivalent of reducing sugars which are indicated by Fehling's solution after boiling with acid. Lenz and several others have examined the value of this determination. The conclusions which are deducible from their experience and our own are that with attention to proper condition to insure complete conversion to dextrose the results are of value, though apt to fall out too low. It has been found desirable in our laboratory to run as a check a parallel determination on a substance of known reduction equivalent. In this way any variation may be detected.

* Fleischer, E., 1884, Zeit. Anal. Chem., 23, 33.

The method of Lenz is described under pepper and its composition. The conditions which we have found most desirable are as follows :

The 2 to 5 grams of the material, usually 4, which should pass an 80-mesh sieve, must be extracted with strong alcohol and with cold water for some time to remove substances not starch which might be acted on by the acid or reduce Fehling's solution. Then, without drying, it is washed off the filter into an Erlenmeyer flask with about 175 c. c. of water and enough strong C. P. hydrochloric acid added, about 25 c. c., to make the liquid 4 to 5 per cent. of acid gas. The flask is then supplied with a condenser and boiled for four hours, or the liquid may be put in a patent rubber-stoppered beer bottle and digested in a steam bath ; but the latter method is not as certain. After the boiling and cooling, the residue is filtered out, the liquid accurately neutralized with sodic hydrate and made up to 500 c. c. It is then titrated as usually, and can be calculated to glucose or starch on any basis desired. Thorough previous extraction and uninterrupted boiling are the two most important conditions. Without extraction the results are most uncertain and unreliable.

Determination of tannin.—The amount of tannin in certain spices, such as cloves and allspice, is quite constant when they are of good quality. Dr. Ellis of Toronto has therefore recommended this as a good means of detecting adulteration in these spices. He has published as yet nothing in regard to his methods of application. In our experiments it has been found to be of some value, but that it is hardly worth while as a mere aid to the detection of adulteration to go so far as the actual determination of tannin, it being quite sufficient to determine the amount of material oxidizable with permanganate which is extracted by water after the careful removal of the oil, &c., by ether. This avoids the tedious use of hide powder, or glue, and furnishes results which are relatively of the same value.

The analyst should therefore prepare himself for carrying out the first part of the modified Löwenthal process, as it is described in Sutton,* and more elaborately in the *Berichte über die Verhandlungen der Commission zur Bestellung einer einheitliche Methode der Gerbstoffbestimmung*, and in *Annales de la Science agronomique*, Tome 1, 1886.

Particular pains should be taken to secure a good article of indigo carmine, as without this the results are unreliable. That recommended by Schröder is manufactured by Gehe & Co., of Dresden, and is known as *Carminum cœruleum*. It should be imported for use.

Without going into particulars as to the reagents employed in the process, which are probably familiar to all, a few words of caution as to detail of manipulation will be of value.

As has been said, preliminary extraction of the material with Squibbs best ether is necessary to remove oil and other substances not tannin, on which the permanganate may act. Ordinary ether will not answer,

* Sutton, Volumetric Analysis, 3d edition, pp. 276.

as it contains so much alcohol and water as to dissolve some of the tannin. The substance freed from ether should be extracted with boiling water, and the extract made up to such dilution that 10 c. c. is equal to about 10 c. c. of the thirtieth normal permanganate solution used. The titration must be performed slowly to insure accuracy, the permanganate being run in at a rate of not more than a drop in a second or three in two seconds. The eye must become accustomed to the bleaching of the indigo and select some one tint of yellow as the end of reaction. It is then possible to obtain duplicates agreeing within .1 c. c. even on entirely different tests, as the following figures show:

Serial No.	C. c. of Perm.	Per ct. of tannin.
4904.	4. 33	22. 46
	4. 3	22. 36
	4. 35	22. 62
	4. 15	21. 58

The results may be calculated to oxygen consumed or to percentage of quercitannic acid, which would not be strictly correct, 1 c. c. of $\frac{N}{30}$ permanganate being equivalent to .0052 grams of quercitannic acid. The results obtained with cloves and allspice will be found under those spices.

Crude fiber.—This is a merely relative determination, as the term crude fiber designates nothing absolute beyond the fact that a certain amount of substance is insoluble in acid and alkali of certain strength after treatment for a definite length of time at a definite temperature. The conditions selected by us are, 2 grams of substance, 200 c. c. of 5 per cent. hydrochloric acid, steam bath two hours, raising the liquid to a temperature of 90° to 95° C., filtration on linen cloth, washing back into beaker with 200 c. c. 5 per cent. sodic hydrate, steam bath two hours, filtration on asbestos, washing with hot water, alcohol, and ether, drying at 120° weighing, ignition and crude fiber from loss in weight. This method agrees practically with that known as the Weende method, and while furnishing results which are of some comparative value, leaves much to be desired. The subject will probably be reviewed soon by the Association of Official Agricultural Chemists of the United States.

Nitrogen and albuminoids.—The methods of determining nitrogen and albuminoids have been discussed and described at length in Bulletin No. 12 of this division. The details of the method of Kjeldahl, as given by Dr. Jenkins, which is the most convenient, are as follows:

Determination of nitrogen by the method of Kjeldahl.

REAGENTS AND APPARATUS.

(1) Hydrochloric acid whose absolute strength has been determined, (a) by precipitating with silver nitrate and weighing the silver chloride, (b) by sodium carbonate, as described in Fresenius's Quantitative Analysis, second American edition, page 680,

and (c) by determining the amount neutralized by the distillate from a weighed quantity of pure ammonium chloride boiled with an excess of sodium hydrate.

(2) Standard ammonia whose strength, relative to the acid, has been accurately determined.

(3) "C. P." sulphuric acid, Sp. Gr. 1.83, free from nitrates and also from ammonium sulphate, which is sometimes added in the process of manufacture to destroy oxides of nitrogen.

(4) Mercuric oxide, HgO , prepared in the wet way. That prepared from mercury nitrate cannot safely be used.

(5) Potassium permanganate tolerably finely pulverized.

(6) Granulated zinc.

(7) A solution of 40 grams of commercial potassium sulphide in one liter of water.

(8) A saturated solution of sodium hydrate free from nitrates, which are sometimes added in the process of manufacture to destroy organic matter and improve the color of the product.

(9) Solution of cochineal prepared according to Fresenius's Quantitative Analysis, second American edition, page 679.

(10) Burettes should be calibrated in all cases by the user.

(11) Digestion flasks of hard, moderately thick, well-annealed glass. These flasks are about 9 inches long, with a round, pear-shaped bottom, having a maximum diameter of $2\frac{1}{2}$ inches, and tapering out gradually in a long neck, which is three-fourth of an inch in diameter at the narrowest part, and flared a little at the edge. The total capacity is 225 to 250 cubic centimeters.

(12) Distillation flasks of ordinary shape, 550 cubic centimeters' capacity, and fitted with a rubber stopper and a bulb tube above to prevent the possibility of sodium hydrate being carried over mechanically during distillation. This is adjusted to the tube of the condenser by a rubber tube.

(13) A condenser. Several forms have been described, no one of which is equally convenient for all laboratories. The essential thing is that the tube which carries the steam to be condensed shall be of block tin. All kinds of glass are decomposed by steam and ammonia vapor, and will give up alkali enough to impair accuracy. (See Kreussler and Henzold, *Ber. Berichte*, *XVII*, 34.) The condenser in use in the laboratory of the Conn. Exp. Station, devised by Professor Johnson, consists of a copper tank supported by a wooden frame, so that its bottom is 11 inches above the work-bench on which it stands. This tank is 16 inches high, 32 inches long, and 3 inches wide from front to back, widening above to 6 inches. It is provided with a water-supply tube which goes to the bottom and a larger overflow pipe above. The block-tin condensing tubes, whose external diameter is $\frac{3}{8}$ of an inch, 7 in number, enter the tank through holes in the front side of it near the top, above the level of the overflow, and pass down perpendicularly through the tank and out through rubber stoppers tightly fitted into holes in the bottom. They project about $1\frac{1}{2}$ inches below the bottom of the tank, and are connected by short rubber tubes with glass bulb tubes of the usual shape, which dip into glass precipitating beakers. These beakers are $6\frac{1}{2}$ inches high, 3 inches in diameter below, somewhat narrower above, and of about 500 cubic centimeters capacity. The titration can be made directly in them. The seven distillation flasks are supported on a sheet-iron shelf attached to the wooden frame that supports the tank in front of the latter. Where each flask is to stand a circular hole is cut, with three projecting lips, which support the wire gauze under the flask, and three other lips which hold the flask in place and prevent its moving laterally out of place while distillation is going on. Below this sheet-iron shelf is a metal tube carrying seven Bunsen burners, each with a stop-cock like those of a gas combustion furnace. These burners are of larger diameter at the top, which prevents smoking when covered with fine gauze to prevent the flame from striking back.

(14) The stand for holding the digestion flasks consists of a pan of sheet-iron 29 inches long by 8 inches wide, on the front of which is fastened a shelf of sheet-iron

as long as the pan, 5 inches wide and 4 inches high. In this are cut six holes $1\frac{1}{2}$ inches in diameter. At the back of the pan is a stout wire running lengthwise of the stand, 8 inches high, with a bend or depression opposite each hole in the shelf. The digestion flask rests with its lower part over a hole in the shelf and its neck in one of the depressions in the wire frame, which holds it securely in position. Heat is supplied by low Bunsen burners below the shelf. Dr. Jenkins has used asbestos paper under the flasks, but finds that with a little care the naked flame can be applied directly to the flask without danger.

THE DETERMINATION.

One gram of the substance to be analyzed is brought into a digestion flask with approximately 0.7 gram of mercuric oxide and 20 cubic centimeters of sulphuric acid. The flask is placed on the frame above described in an inclined position and heated below the boiling point of the acid for from five to fifteen minutes, or until frothing has ceased. The heat is then raised till the acid boils briskly. No further attention is required till the contents of the flask has become a clear liquid, which is colorless or at least has only a very pale straw color. The flask is then removed from the frame, held upright, and while still hot, potassium permanganate is dropped in carefully and in small quantity at a time till after shaking the liquid remains of a green or purple color. After cooling, the contents of the flask are transferred to the distilling flask with water, and to this 25 cubic centimeters of potassium sulphide solution are added, 50 cubic centimeters of the soda solution, or sufficient to make the reaction strongly alkaline, and a few pieces of granulated zinc. The flask is at once connected with the condenser and the contents of the flask are distilled till all ammonia has passed over into the standard acid contained in the precipitating flask previously described and the concentrated solution can no longer be safely boiled. This operation usually requires from twenty to forty minutes. The distillate is then titrated with standard ammonia.

The use of mercuric oxide in this operation greatly shortens the time necessary for digestion, which is rarely over an hour and a half in the case of substances most difficult to oxidize and is more commonly less than an hour. In most cases the use of potassium permanganate is quite unnecessary, but it is believed that in exceptional cases it is required for complete oxidation, and in view of the uncertainty it is always used. Potassium sulphide removes all mercury from solution and so prevents the formation of mercurio-ammonium compounds which are not completely decomposed by soda solution. The addition of zinc gives rise to an evolution of hydrogen and prevents violent bumping. Previous to use the reagents should be tested by a blank experiment with sugar, which will partially reduce any nitrates that are present which might otherwise escape notice.

This method cannot be used for the determination of nitrogen in substances which contain nitrates or certain albuminoids.

In case non-albuminoid nitrogen is to be determined reference can be made to Stutzer and Ladd.*

These methods of analysis are suitable to all the spices and have been used with them. They are nothing but general processes, and are dependent for their value on uniformity in the way they are carried out and the manner in which peculiarities of proximate composition in different spices are considered in drawing conclusions. Determinations of particular substances, such as piperine, require, however, modifications, which must be described when discussing the analyses of each spice.

* Rept. anal. Chem., **5**, 162, 163; Abs. Bér., **19**, 1885; and Ladd Rept. of New York Agric. Exp. Sta., 1886.

MUSTARD.

Mustard of commerce is the seed, whole or ground, of several species of the genus *Brassica*, cruciferous plants which grow wild and are cultivated under very various conditions. The two common varieties are the black or brown mustard, which has a very small seed and furnishes the most aroma, and the white, which is two or three times as large, often used in the whole condition in pickles and ground, either by itself or oftener in mixture with the brown seed, for the purpose of obtaining the desirable qualities of both.

In the ground mustard is found only the interior of the seed and small portions of the husks which have escaped the operation of bolting, which is always employed to remove the coarse fragments. The presence of these particles from their characteristic structure enable us to recognize the source from which the flour is derived and to detect the use of the mustard hulls as adulterants of other food materials. The husk of white mustard is represented, after a drawing by Schimper, in Fig. 10.

The outer colorless epidermis consists of angular plates or hexagonal tabular cells with a center of different brilliancy. They swell up and become slimy in water and must therefore, be observed in glycerine. At the best it requires some manipulation to see it well, and it is far less prominent in the brown seed. The next coat, denominated the subepidermal, is not prominent and can only be seen at all easily in the white seed.

The third layer is an important one. In it is found the coloring matter of the brown seed, and its absence is the cause of the lack of color in the white variety. Fragments of this layer are common in ground mustard. It is distinguished by the thick or colorless brown cell walls and their irregular dotted appearance. Once examined it will be readily recognized under other circumstances, as, for example, when the hull is used as an adulterant of pepper.

Between this layer and the next are some unimportant and difficultly discernible cells carrying in the brown seed some color.

Within these comes the important layer denominated the inner tunic by Hassall and the plasma layer by Schimper. It separates readily from the other parts of the husk and is often found by itself in the ground mustard. As its contents are broken up by water or chloral hydrate, glycerine or oil must be used as a mounting medium. The cells of which the layer consists are large, and with their contents are similar to the embryous envelope or false gluten cells of wheat, to which they correspond. They are much alike in both white and brown mustard. These structures are diagrammatically represented in Fig. 7.

From the character of the exterior layer and the lack of color in the third layer, as well as minor differences which are not describable, but will appear to the patient investigator, it is always possible to tell

whether the flour of mustard is a mixture of the two varieties or from one alone. Mounts in chloral hydrate, to a certain degree, are useful for adding to the transparency of the substance. The interior of the seed is made up of small soft parenchyma cells containing the oil and other constituents of the mustard, but without any trace of starch. For this reason the presence of starch is a certain indication of the addition of some diluent of a farinaceous nature. Simple treatment with iodine will therefore reveal the presence of wheat flour, which is a common adulterant of this condiment. The white color of the flour of course reduces the yellow color of the mustard, and it is usual, therefore, to restore the tint by either *turmeric* or *Martin's yellow*.

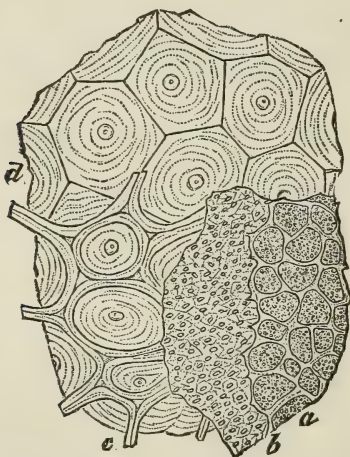


FIG. 10. Husk of white mustard. *a*, *b*, plasma layer; *c*, sub-epidermal; *d*, epidermis.

The former can be detected by a mechanical separation or a brown coloration with ammonia or by the peculiar color cells which it contains and the starch granules of the arrowroot class. The latter is not sufficient in amount to be confused with potato starch or that of flour. It has been already described.

Martin's yellow can be identified by extraction with cold 95 per cent. alcohol and examination, after evaporation of the solvent, as suggested by Waller and Martin, and this coloring matter seems to be often used, and cannot be pronounced as harmless as turmeric.

The substances mentioned are the common adulterants of mustard, in fact, so common that they have been accepted as necessary dilu-

ents, being considered desirable for toning down the pungency and adding to the keeping qualities of the ground material. Of late years, however, a reaction has taken place, and it is now possible to find brands of pure ground mustard.

In most of the samples which have come into our hands for examination flour and coloring matter are the only foreign substances which have been met with. From the investigations of foods chemists abroad, it would appear on the authority of Hassall and others that other species of mustard seed, rape seed, cayenne pepper, ginger, potato flour, rice, pea flour, seed meals, and several mineral substances are frequently found in the mustards of commerce, a conclusion which we have found justified by the presence in some of the lower-grade mustards which have come into our hands of yellow corn, ginger, mustard hulls, gypsum, and sand. The presence of these adulterants, which is only too common in the cheaper article when sold in bulk and under no brand, can be determined by mechanical means and by their structure, which is quite different from that of the mustard, and by the starches, which characterize some of them, as already explained.

While the adulterants of mustard, therefore, are, owing to the characteristic structure of the seed, easily detected with the microscope, in cases where there is doubt, or where further information is desired as to the probable proportion of diluent, recourse must be had to determinations of the chemical constituents of the sample.

CHEMISTRY OF MUSTARD.

Several investigators have made proximate analyses of mustard. Hassall has collected the following in regard to its composition, and has also made several analyses of pure and adulterated samples :

Of these seeds no very complete quantitative analyses have as yet been made, although many highly important particulars have been ascertained respecting their composition; thus black or brown mustard, as it is now generally named, consists for the most part of *fixed oil*, *myronic acid* $C_{10}H_{19}NS_2O_{10}$, which is combined with potash, forming a *myronate of potash*, and which acid is converted into the volatile oil of mustard or *sulphocyanide of allyl* C_4H_5NS or $\left. \begin{matrix} CN \\ C_3H_5 \end{matrix} \right\} S$ through the agency of the *myrosin*, another constituent of brown mustard, when the two are brought into contact through the medium of water, *vegetable albumen*, a *bitter principle*, a little *gum* and *sugar*, a *peculiar green substance*, *cellulose* and *mineral matter*.

White mustard differs essentially in its composition from brown; it also contains *fixed oil*, but in lieu of myronic acid, convertible as described into the volatile oil of mustard, it contains a non-volatile, bitter and acrid salt, termed *sulphocyanide of synapine* ($C_{17}H_{24}N_2SO_5$ or $C_{16}H_{23}NO_5CNHS$), *myrosin*, *gum*, *cellulose*, and *mineral matter*.

Now it is on the volatile oil and the acrid and somewhat bitter salt that the pungency and acidity of mustard depends, and hence we see a strong reason why in the mustards of commerce the farina of the two species should be blended together; of the two active principles the volatile oil is by far the more important, and hence the seed of the brown mustard possesses the greatest commercial value. It should be stated that Henrie and Garot affirms that brown mustard contains the acrid principle as well as the white; this statement we have been able to verify as shown specially by the action of nitric acid, caustic potash, and ferric chloride on the alcoholic extract.

The acrid principle of white mustard appears to possess but little stability, and although it is stated by V. Babo to bear a temperature of $130^{\circ}\text{C}.$, we find that it is readily affected by heat, and that it is not safe to evaporate the alcoholic solution containing it at a higher temperature than about $30^{\circ}\text{C}.$ If subjected to a much higher temperature it quickly loses its acidity and acquires a bitter caramel-like taste.

Of neither brown nor white mustard had any percentage analysis been given until those made and published by ourselves in an article on mustard and its adulterations, in "Food, Water, and Air," for February, 1874; and in the few cases in which the quantities of any of the constituents are stated, they vary greatly according to different observers. Thus, according to Pereira, the *fixed oil* forms about 23 per cent. of the seeds of black mustard, while Watts puts the yield at 18 per cent. only, but white mustard seed, he says, furnishes 36 per cent. The *volatile oil* amounts to 0.20 per cent., according to Boutron and Robiquet; 0.55 per cent., according to Aschoff, and 0.50 per cent. according to Wittstock; all of which quantities are much below the mark, as will be seen hereafter. Now, as will be shown presently, there is little or no difference in the amount of fixed oil furnished by the two descriptions of mustard, that obtained by me from the farina of brown mustard reaching 35.701 per cent., and that from the white mustard 35.768 per cent. Again it is shown by the analyses given below that the volatile oil occurs in much larger quantities than those enumerated above, the amount which we have obtained from one sample being no less than 1.271 per cent.

Of both brown and white mustard we append the following original percentage analyses, first published in the article referred to:

BROWN MUSTARD FARINA.

	Per cent.
Water.....	4.845
Fixed oil.....	35.701
Myronic acid.....	4.840
Myrosin and albumen.....	29.536
Acrid salt.....	3.588
Cellulose.....	16.765
Ash.....	4.725
	100.000
Volatile oil.....	1.271
Nitrogen.....	5.068
Sulphur.....	1.413

The oil extracted by ether from the brown seed is of a bright and beautiful emerald green color, owing to the presence of the peculiar green principle described as one of its constituents. So deep and remarkable is the color of the oil that it would be easy, by means of a graduated scale of tints, to determine with very tolerable certainty the percentage of brown mustard contained in any samples of mixed mustard.

WHITE MUSTARD FARINA.

	Per cent.
Water.....	5.360
Fixed oil.....	35.768
Acrid salt.....	10.983
Myrosin and albumen.....	27.484
Cellulose.....	16.295
Ash.....	4.110
	100.000
Nitrogen.....	5.285
Sulphur.....	1.224

These analyses, whether regarded from a scientific or practical point of view, are possessed of much interest.

The small quantity of sugar found in mustard would, from the method of analysis pursued, be included under the bitter principle and the gum with cellulose.

Of the methods of analysis, Hassall writes:

Estimation of myronic acid.—Myronate of potash decomposes, under the influence of the nitrogenous matter contained in brown mustard, into volatile oil, glucose, and acid sulphate of potash. The quantity of each of these products of decomposition gives, therefore, by simple calculation, the quantity of myronic acid. One hundred parts of this acid yield 23.85 parts of volatile oil. From 40 to 50 grams of the mustard farina are placed in a flask of about one-half liter capacity; 250 c. c. of tepid water are poured over it, the flask closed with a cork, and the whole is well shaken. After twenty-four hours' standing the flask is connected with a Liebig's condenser, and its contents are heated to boiling. Into the receiver 30 c. c. of strong ammonia are poured, and the end of the condenser is dipped below the surface of the liquid. Water and the volatile oil pass over, the latter at first floating in the shape of oily drops on the surface of the liquid, which soon sink to the bottom, especially when the liquid is gently agitated. When the distillation is finished, which is the case when no more oil globules pass over, the receiver is closed with a cork and allowed to stand twenty-four hours. At the end of that time all the oil is dissolved and is now contained in the liquid in the form of thiosinamin. This solution is evaporated on the water bath in a weighed platinum basin, the residue dried and weighed. The quantity of thiosinamine obtained, minus one molecule of ammonia, represents the amount of volatile oil.

Estimation of the myrosin or albumen and of the sulphocyanide of sinapin.—The total amounts of nitrogen and sulphur contained in the mustard are next ascertained. The former by combustion with soda-lime in the well-known manner, the latter by deflagration of the mustard and oxidation of its sulphur in a mixture of nitrate of soda and carbonate of potash. The fused mass is dissolved in water or dilute acid, and the sulphuric acid contained in the solution is estimated by means of chloride of barium. From these data the amounts of the myrosin and of the sulphocyanide of sinapin, the acrid principle, are thus calculated; as much sulphur and nitrogen are first deducted from the totals of these substances obtained as is contained in the quantity of myronic acid previously determined. Next, the whole of the remaining sulphur and as much of the nitrogen as is required are then calculated into the acrid principle; lastly, the surplus nitrogen is calculated into myrosin, which has the same formula as vegetable albumen. But now, having got approximately the amounts of the acrid principle and of the myrosin, a further calculation has to be made, since myrosin contains about 1 per cent. of sulphur. This has to be deducted from the total acrid principle, a corresponding quantity of nitrogen being in its turn calculated into myrosin. By those acquainted with algebra it will readily be perceived that a more precise calculation may be made, but the results would not, even then, differ to any practical extent.

Acting on this method, Hassall made several analyses of genuine mustards of the trade and also of adulterated articles, which are here presented, merely dropping the third place in decimals, which is of no value.

Analyses of genuine mustard.

[Hassall, pp. 514-516.]

	Genuine mustard.	Genuine double superfine.	Genuine superfine.	Genuine Fine.	Pure.	House- hold.
Water	5.70	5.16	5.59	5.68	5.08	5.29
Fixed oil	36.49	35.94	34.71	35.24	33.98	36.75
Myronic acid	2.70	2.21	1.97	.92	.96	1.72
Myrosin and albumen	31.69	27.36	31.02	27.90	27.62	8.75
Acrid salt and bitter principle..	5.72	9.09	7.10	10.06	11.26	27.48
Cellulose	13.37	15.58	15.29	15.55	16.81	3.69
Ash	4.33	4.66	4.32	4.65	4.29	16.32
	100.00	100.00	100.00	100.00	100.00	100.00
Oil of mustard71	.58	.52	.24	.25	.45
Nitrogen	5.34	5.05	5.46	5.16	5.21	5.03
Sulphur	1.31	1.42	1.25	1.30	1.40	1.31

Analyses of mixed and bulk mustard.

	Double super- fine.	Fine.	Super- rior.	Suerls.	Alexan- der.	Lind- sey.	Gilbert.	Good- man.	Clark.
Water	4.94	6.51	4.97	8.94	8.34	8.87	6.28	8.95	9.58
Fixed oil	27.52	23.16	25.17	24.88	29.60	21.54	22.06	26.90	18.31
Myronic acid	3.14	1.36	1.20	1.57	1.92	.98	1.13	1.82	.39
Acrid principle	1.85	5.81	4.31	6.45	3.15	6.21	4.25	5.18	7.03
Myrosin	23.16	19.50	23.24	14.48	13.89	21.76	15.30	15.58	20.82
Wheat flour and turmeric.	22.99	27.20	25.82	33.81	30.52	25.21	38.82	30.56	32.81
Cellulose	13.05	12.84	11.50	7.08	8.99	11.69	8.41	7.27	8.65
Ash	3.35	3.62	3.79	3.79	3.59	3.74	3.75	3.74	2.41
Volatile oil85	.36	.32	.41	.50	.26	.30	.48	.10
Nitrogen	4.24	3.85	4.07	3.34	3.16	4.30	3.46	3.37	3.33
Sulphur95	.96	1.06	1.00	.90	.94	.82	.94	.91

"Of the first six analyses of genuine mustards" Hassall says that they "prove two things; first, that all the samples are genuine; this is shown by the quantities of fixed oil, nitrogen, and sulphur obtained; and that they consist of mixtures of the two mustards in different proportions, the higher qualities containing larger proportions of the brown mustard; that this is so is demonstrated by the different quantities of volatile oil obtained."

In the analyses of the adulterated mustards allowance in the calculation was made for the nitrogen of the wheat flour.

Hassall says:

From an examination of the foregoing analyses it is apparent that genuine *brown* mustard should contain about 36 per cent. of fixed oil, at least 1 per cent. of volatile oil of mustard, about 4 per cent. of acrid principle, and that it should furnish about 1.5 per cent. of sulphur and 5 per cent. of nitrogen; that genuine *white* mustard should yield about the same amount of fixed oil, over 10 per cent. of acrid principle, and nearly the same amount of nitrogen and sulphur as the black; that the composition of genuine mustards, which are made up in various proportions of brown and white mustard seed, differs according to the quantities of each kind present, the relative proportions being determinable by analysis with considerable precision; that in the mixed or adulterated mustards the proportions of fixed and volatile oil, of nitrogen, and sulphur are all much reduced, according to the extent of the admixtures, these consisting in the mustards now reported upon in all cases of wheat flour and turmeric.

Thus the fixed oil was reduced in one of the samples from 36 per cent., the normal amount, to about one-half or 18 per cent., the volatile oil to 0.1 per cent., and the nitrogen to 3.32 per cent., while in another sample the sulphur was as low as 0.81 per cent. The amount of wheat flour and turmeric varied from 22.91 per cent. to 38.82 per cent., that is to say, from one-fourth to one-third of the article.

These results furnish an excellent basis for the examination of the mustards met with in our country. In addition, however, we have the work of several other investigators. C. H. Piesse and Lionel Stansell* have given their results of the analyses of several samples of pure farinas and their ashes, largely after the method of Hassall. Blyth copies them,† and adds several pages on the chemistry of mustard and its adulterations, adding nothing new to what has been quoted from Hassall, with the exception of a formula for calculating the percentage of added flour in mixtures from the amount of fixed oil found. He says :

Estimation of fat or oil.—This is particularly useful when wheat starch is the adulterating agent. Wheat flour does not contain more than 1.2 to 2.1 per cent. of oil; mustard, on the other hand, from 33.9 to 36.7 per cent. A weighed portion of the previously dried samples may be placed in an extraction apparatus, and from the oil found the following formulæ will serve as a guide to the amount of flour present :

x = amount of mustard, y = amount of oil found.

$$\frac{33.9 x}{100} + \frac{1.2 (100-x)}{100} = y \qquad \frac{36.7 x}{100} + \frac{2 (100-x)}{100} = y$$

according as the greater or less amount of oil is taken as being present in the pure farina and the flour.

Accepting the mean for mustard and 2.0 per cent. as the proper figure for flour, the formula would read more conveniently, it would seem,

$$x = \frac{y-2}{1.333}$$

where $x=100$ when the mustard is pure. Of the amount of ash Blyth says :

"The total ash of dried mustard averages 5 per cent. The highest number the writer has obtained is 5.3 per cent.; the lowest 5.088 per cent. Of this ash 1.2 at least is soluble in water; in other words, the ash of mustard consists of 39 parts per cent. soluble, 70 parts per cent. insoluble in water. It hence follows that if found above 5.5 per cent. mineral matters of foreign origin are present; if below 4 per cent. it is an indication of some organic adulterant."

Albert R. Leeds and Edgar Everhart‡ have taken up Hassall's and Blyth's work and shown that the latter's formula for calculating added flour from the percentage of oil found will not serve in all cases, as it is not uncommon to express some of the oil from the seed before grinding, to adulterate with oil cake or seeds, or to add cheap oils to the flour used as a diluent to cover any deficiency. They also analyzed a sample of pure farina of brown mustard according to a method devised by themselves as a modification of Hassall, in which the determinations should be direct instead of calculated. Briefly, it is as follows :

* Analyst, **5**, 161-165, 1880.

† Foods and their adulterations, 485-486.

‡ Zeit. anal. Chem. **21**, 389-394, 1882.

METHOD OF LEEDS AND EVERHART.

Moisture and ash are determined as usual; oil, in a portion of mustard dried at 105° , by ether in an extraction apparatus, with subsequent drying at 100° . From the dried residue the *sulphocyanide of sinapin* and the *myronate of potassium* are extracted in a similar way by 50 per cent. alcohol. The extract is dried, weighed, and ignited, and from the sulphate of potash in the ash the myronate is reckoned and the sulphocyanide obtained by difference. The residue, containing *myrosin* and *cellulose* and a little coloring matter, is freed from alcohol and treated with one-half per cent. sodic hydrate solution. The washed residue is weighed and ignited for cellulose. The filtrate containing all the myrosin is nearly neutralized with dilute hydrochloric acid, 50 c. c. of Ritthausen's copper sulphate solution added, and then dilute sodic hydrate to near neutrality. The heavy green myrosin copper compound is filtered off, dried at 110° C., weighed, and ignited. The difference is the myrosin.

Analyses carried out after this method in triplicate are as follows:

	Per cent.	Per cent.	Per cent.
Water.....	6.78	6.90	6.82
Myronate of potash.....	.61	.61	.72
Sulpho-cyanide of sinapine.....	10.97	11.19	11.21
Myrosin.....	28.45	28.70	28.80
Oil.....	29.22	29.21	29.19
Cellulose (by difference).....	20.24	19.55	20.06
Ash.....	3.73	3.84	3.70

The amounts of nitrogen and sulphur in this mustard were:

Nitrogen	5.337
Sulphur	1.489

Calculated from these figures according to the method of Hassall would be found the following:

Myronate of potassium61
Sulphocyanide of sinapine	10.71
Myrosin	28.52

Showing a close agreement with the direct determinations.

Direct methods are, however, usually preferable and this would, no doubt, be a good one were it not that dilute alcohol in the case of admixture of flour would dissolve so much of the albuminoid matter and ash of the latter as to invalidate the determination of myronate. It is therefore open to criticism.

E. Waller and E. W. Martin* in 1882 made an examination of mustards manufactured and sold in New York City, attention being paid to moisture, oil, and soluble and insoluble ash. They also examined mustard pastes, and compared their results with some pure mustards from the English market. Their analyses are as follows:

* Analyst 9, 166-170.

Dry mustard manufactured and sold in New York City.

No.	Moist- ure.	Fixed oil.	Soluble ash.	Insolu- ble ash.	Total.	Coloring.	Remarks.
197	6.15	21.17	.30	5.54	5.84	Martin's yellow..	Contains starch.
204	8.03	12.79	1.39	5.39	6.78	Turmeric	Contains starch and Ca SO ₄ .
206	7.35	12.54	.23	4.69	4.92do	Ash fixed starch.
207	8.23	8.42	.15	1.90	2.05	Martin's yellow...	Contains starch.
208	8.50	10.92	2.90	13.15	16.05	Turmeric	Contains starch, Ca SO ₄ .
209	7.24	6.81	.10	3.55	3.65do	Contains starch
213	7.65	13.32	.64	5.17	5.81	Martin's yellow...	Contains starch, ash-fused.
214	7.60	7.74	1.53	1.69	3.22	Turmeric	Contains starch.
215	7.15	9.02	.20	2.91	3.11do	Contains starch.
216	5.45	20.57	.15	5.12	5.27do	Contains starch.
217	6.50	8.59	1.52	5.65	8.17do	Contains starch, Ca SO ₄ .
218	8.45	14.59	2.15	6.65	8.80do	Contains starch, Ca SO ₄ .
219	6.62	22.56	1.62	4.86	6.48do	No starch.
294	9.86	6.21	1.16	3.54	4.70	Martin's yellow...	

Mustard paste, German mustard manufactured in New York City.

No.	Moist- ure.	Acetic acid.	Oil.	Other organic sub- stance.	Soluble ash.	Insolu- ble ash.	Total.	Common salt.	Oil on dry sub- stance.	Metallic copper.
221	77.02	2.76	2.55	14.18	2.51	0.93	3.49	2.11	24.98	.001
222	81.52	1.98	3.50	10.67	1.77	.56	2.33	1.63	21.24	Trace.
237	79.62	2.43	3.90	12.60	2.52	.97	3.49	19.51	.009
242	76.54	3.69	4.57	11.53	2.69	.98	3.67	1.86	23.14	.003
244	81.45	2.94	3.73	9.09	2.14	.65	2.79	1.77	22.44	Trace.

Mustard flour (bolted) purporting to be pure.

No.	Moist- ure.	Oil.	Soluble ash.	Insolu- ble ash.	Total.	Remarks.
201	6.10	26.42	.21	5.92	6.21	New York manufacture.
220	5.50	25.70	.86	4.80	5.66	New York manufacture, Trieste and Bombay seed, mixed.
273	4.85	36.67	.175	3.725	3.900	English samples, whole seed.
274	4.75	41.70	.125	4.425	4.550	English samples, brown seed, ash-fused.

Ground mustard seeds.

No.	Kind of seed.	Moist- ure.	Oil.	Soluble ash.	Insolu- ble ash.	Total.
	American market:					
231	Bombay	7.52	36.96	1.25	4.37	5.62
232	Trieste	6.35	36.45	.70	3.70	4.40
233	California yellow	4.95	34.00	.50	4.40	4.90
234	English yellow	6.10	35.46	.25	4.55	4.80
	English market:					
271	White	7.10	34.45	.70	3.90	4.60
272	Brown	7.30	34.71	.85	3.90	4.75

As comment they say :

The results obtained for oil on Nos. 201 and 220 led to inquiry, the result of which was the discovery that it is the regular practice of the mustard manufacturers here to express a portion of the oil from the ground mustard seed before working it up into the condiment sold as mustard. In these samples, as well as in No. 219, which was sold under guarantee of being pure mustard without admixture, no starch, coloring material, or other material known to be foreign to the mustard seed was found.

If we calculate, then, that these mustards have been made up from mustard flour similar to 201 and 220, and containing 25 per cent. of oil, by multiplying the percentages of oil given in the table by four, we shall get approximately the proportions of mustard flour present, in percentages.

These investigators also examined a specimen of English mustard which was mixed with starch, and yet contained a normal amount of oil which, on extraction, was decidedly more fluid than mustard oil. This points to the truth of the assertion that the extraction of the mustard oil is often covered by the addition of oil of inferior character.

The common use of Martin's yellow (Dinitronaphthol) as a coloring matter is startling, as it can hardly be anything but injurious. They detected it by extracting the flour with cold, strong alcohol, evaporating, taking up with water and dyeing wool with it. Crystals, however, could not be obtained, but the authors came into possession of a sample of the coloring matter which was analyzed.

In Canada, large numbers of samples of mustard have been examined with results which have already been quoted, showing that the manufacture of mustard from mustard cake and the addition of farinaceous matter is as common there as elsewhere. In the report for 1885, out of fifty-one specimens, only 10 contained over 25 per cent. of fixed oil and no starch, and but three contained over 30 per cent. oil, as they should, if of best quality. The chief analyst, however, is of the opinion that the removal of the oil is an advantage, as the flour will keep better and be as pungent without it.

With the information of the nature which has been given a number of samples of mustard purchased in the open market in Washington, some of low grade obtained direct from Baltimore spice mills, and others in the whole seed from the importers and dealers, have been examined microscopically and chemically. They may be described as follows:

Serial No.	Price $\frac{1}{2}$ pound (retail).	Remarks.
	<i>Cents.</i>	
4510	White seed.
11	30	Guaranteed pure.
22	20	Ground in District of Columbia.
27	20	Ground in Baltimore.
32	30	English brand.
36	36	Ground in Baltimore.
42	30	English brand.
50	20	
51	20	Ground in New York.
4241	Mohawk, N. Y.
71	Extra American, Baltimore, Md.
72	London, strong, extra, Baltimore, Md.
4971	30	Ground in Baltimore; extensively advertised as pure.
4885	White-seed flour, ground in laboratory.
86	White-seed husk, ground in laboratory.
99	California yellow mustard seed.
4900	California brown mustard seed.
1	English yellow mustard seed.
2	Trieste brown mustard seed.

The analytical determinations gave the following results :

Analyses of mustard.

[Whole-seed flour.]

Serial No.	Source.	Weight of 100 seeds, in grams.	Quality.	Water.	Ash.	Volatile oil.	Fixed oil.	Starch.	Crude fiber.	Albuminoids.	Undetermined.*	Total.	Nitrogen.
4510	White seed635	5.57	4.29	.97	33.56	.00	5.40	28.88	21.33	100	4.62
4885	White flour	3.33	5.23	1.84	34.83	.00	9.05	25.56	20.16	100	4.09
4886	Seed husk480	6.17	4.99	.55	28.12	.00	9.50	23.44	27.23	100	3.75
4899	California yellow460	4.83	5.96	1.27	31.96	.00	8.50	31.13	16.35	100	4.98
4900	California brown135	4.11	4.88	1.35	36.63	.00	16.18	24.69	12.16	100	3.95
4901	English yellow419	3.11	4.07	2.06	31.51	.00	6.90	30.25	22.10	100	4.84
4902	Trieste brown423	4.62	5.61	.63	39.55	.00	10.84	25.88	18.87	100	4.14

[Commercial mustard flour.]

4511	(?)	Colored	5.97	6.55	.43	18.16	.00	7.35	37.44	24.10	100	5.95
4522	District of Columbia	Adulterated	9.38	7	.24	11.20	21.15	2.95	20.63	27.04	100	3.30
4527	Baltimore	9.73	4.40	.80	9.85	29.14	2.10	13.31	30.37	100	2.13
4532	English	5.84	4.90	2.11	30.84	7.26	4.63	25.88	18.54	100	4.14
4536	Baltimore	6.60	9.70	.50	14.72	9.00	3.75	14.50	41.23	100	3.92
4542	English	3.25	3.65	2.01	32.26	8.50	14.98	25.19	10.16	100	4.03
4550	(?)	5.80	3.15	.37	7.89	40.50	2.43	14.38	25.46	100	2.30
4551	New York	6.70	1.90	1.31	6.50	45.00	2.97	13.63	21.99	100	2.18
4871	Baltimore	4.57	3.24	2.02	6.77	31.50	2.90	20.31	28.60	100	3.25
4872	Baltimore	7.25	3.35	2.32	5.54	34.63	1.23	19.63	26.05	100	3.14
4971	Baltimore	6.03	5.98	.65	19.46	5.70	3.48	33.06	25.64	100	5.29

Abnormal figures in full-faced type.

DISCUSSION OF THE ANALYSES.

The results obtained with pure seed, ground in the laboratory, show that flour of mustard is fairly constant in its composition.

Water is present in small amount, as is generally the case in oil seeds, varying between 3 and 7 per cent. Hassall found from 5.16 to 5.70, and Waller and Martin 7.52 to 4.95.

Ash is quite constant between 4 and 6 per cent., so that the presence of foreign mineral matter is readily detected. Blyth places the variation between 5.1 and 5.3, Waller and Martin between 4.40 and 5.62, while in ground samples it falls as low as 2.05, Waller and Martin, or 1.90, our own results, owing to the addition of organic adulterants, such as wheat flour, or rises to 16 per cent. from the addition of gypsum. The determination is therefore an extremely valuable one.

Volatile oil is present naturally in the seed in but small amount. The percentage we have found to be rather variable, as much as 2.06 having been found in an English yellow seed and as little as .55 in another. Hassall found from .71 to 24 per cent. Its presence is not of importance.

Fixed oil is one of the most prominent constituents of the seed, varying in amount from 31 to 37 per cent. Waller and Martin give results

varying from 34 to 37, and Hassall 34.71 to 36.49. It has become an almost universal practice, however, to express a portion of the oil, so that in good flour as little as 18 per cent. has been found.

Starch is entirely absent, a contrast to the cereal grains and many other seeds. Its addition is of course common.

Crude fiber varies in a way dependent on the method of milling and of determining its amount. Our samples were found to contain much more than the best flour of commerce, as our means of separating the husk are imperfect. With careful milling not more than 6 to 7 per cent. should be present, according to modern chemical methods. Hassall, on the other hand, found from 13 to 17 per cent.

Albuminoids make up a large part of the seed, varying from 25 to 32 per cent. Anything below 20 per cent. points to dilution with material poor in nitrogen.

The *undetermined* matter consists of gum and some unidentified substances soluble in alcohol, whose estimation is of no particular value as a means of detecting adulteration.

Our results, as a whole, agree closely with those of other investigators, so that for general reference the following standard may be used:

	Per. cent.
Water	3 to 7
Ash	4 6
Volatile oil	$\frac{1}{2}$ 2
Fixed oil:	
When from entire seed.	31 37
When from cake.....	16 18
Starch	None.
Crude fiber	5 18
Albuminoids	25 32

When, however, the flour is ground from cake all the percentages may be somewhat increased.

As compared with the pure mustards the flours of commerce give results which show at once the universal extent of the adulteration which takes place, aside from any microscopic examination.

4511, purchased in Washington but ground elsewhere, has been deprived of nearly half of its fixed oil, that is to say, is ground from mustard cake. It is the only specimen which contains no farinaceous material, and is higher in albuminoids than any of the pure seeds or flours. This is due to the fact that abstraction of the oil raises the relative percentage of albuminoids, and this is not reduced at all by the addition of any diluent. The amount of fiber is relatively high for the same reason. It contains also a very small amount of mineral adulteration. The microscope shows the presence of turmeric as coloring matter.

This brand is perhaps the purest met with, as its only defects are lack of oil which has been considered not a loss, the small amount mineral matter, and the presence of a little coloring matter.

4522, represented by two different specimens, has, in one case, that analyzed, been deprived of more oil than the preceding, has a considerable amount of gypsum in its ash, and 21 per cent. of starch in the shape of wheat flour. The relative percentages of fiber and albuminoids are thereby reduced somewhat, since 21 per cent. of starch would correspond to about 30 per cent. of flour. Color is given by turmeric. In the other case the changes were similar, except that no turmeric was added, but large amounts of white mustard hulls.

4527, ground in Baltimore, consists to a large extent of flour, between 40 and 50 per cent., and is made from mustard cake, the oil having been removed. The color has been restored by turmeric. The sample leaves on sieving quite a large amount of husky and fibrous matter contaminated with wheat flour and turmeric. The husks are not those of the original mustard, and have not been identified, but seem to resemble the exterior coats of ginger, and may represent an addition of spent ginger or ginger tailing.

4522, sieving leaves white mustard hulls and yellow corn meal, but no color; wheat flour is also present.

4551. This sample contains turmeric, wheat flour, and salt.

4532. An English brand, is made from whole seed and is diluted with but little flour and color, about 10 per cent. of the former.

4536 is made from mustard cake, contains a large amount of gypsum and some starch, and is colored (with turmeric). The large amount of undetermined matter would point also to the presence of other adulterants not identified.

4542 resembles the other English brand examined, 4532 being only altered by the addition of a little flour and color.

4550 and 4551 are perhaps the worst samples we have examined. They are made from cake, contain no mineral adulterants, but are more than half flour, and are of course much colored with turmeric. They were purchased from the same grocery. One also contained salt, detected by sifting.

4871 and 4872 are similar and but little superior. They were obtained directly from a Baltimore mill in a lot of spices, which were all adulterated.

4971 has been much advertised as quite pure, but was found to contain sand and flour, but no color. It is made, as usual, from seed cake. From these samples we learn the quality of the ordinary flour mustard of the groceries. It is not good, and certainly demands reform.

PEPPER.

The ordinary black and white peppers of commerce are the fruit of the true pepper plant, *Piper nigrum*, which grows in the East and West Indies. Red pepper, or cayenne, is not a pepper, but the fruit of several species of *Capsicum*. Under the title of "pepper," therefore, attention

will be confined to the genus *Piper*, reserving for a separate chapter the remaining substances which are commonly mis-called peppers.

The pepper plant is a perennial climbing shrub, with a small, round, sessile, fleshy fruit, which grows spontaneously on the Malabar coast, and whose culture has been extended to Siam, Hindostan, Indo-China, Malacca, Singapore, Penang, Ceylon, Sumatra, Java, Borneo, and the neighboring islands, and to a small extent in Guiana and Cayenne.

The greatest production is in Sumatra, and the ports of export are principally Singapore and Penang, the Malabar pepper coming from Tellicherry. Our imports are principally through England, and not direct, and it seems that, in England at least, it is customary to mix peppers of different origin in grinding, taking Malabar for weight, Penang for strength, and Sumatra for color.

Of the other characteristics and history of pepper a most complete account may be found in "Flückiger and Hanbury's Pharmacographia."* Of its preparation for the market these authors say :

When one or two berries at the base of the spike begin to turn red the whole spike is pinched off. Next day the berries are rubbed off with the hands and picked clean, then dried for three days on mats or on smooth, hard ground or on bamboo baskets near a gentle fire.

As thus prepared it is the black pepper of the trade.

When the berries are allowed to ripen, and the black outer pericarp is removed on drying, they are known as white pepper.

The grains of white pepper are of rather larger size than those of black, and of a warm, grayish tint. They are nearly spherical or a little flattened. At the base the skin of the fruit is thickened into a blunt prominence, whence about twelve light stripes run meridian-like toward the depressed summit. If the skin is scraped off the dark-brown testa is seen inclosing the hard, translucent albumen. In anatomical structure, as well as in taste and smell, white pepper agrees with black, which, in fact, it represents in a rather more fully-grown state.

A study of the structure of black pepper will, therefore, furnish every information in regard to the white. There are also two species of *Piper* which furnish a berry used in a similar way to that of *Piper nigrum*. They are known as long pepper, *Piper longum* and *Piper officinarum*. Their structure is similar to that of the common pepper, with some characteristic differences. It is difficult to say how far they are an article of commerce in this country. They come principally from Penang and Singapore, being brought from Java and other places.

The structure of black pepper is described as follows in the work last quoted :

The small, round berry-like fruits grow somewhat loosely to the number of twenty to thirty on a common pendulous fruit stalk. They are at first green, then become red, and, if allowed to ripen, yellow, but they are gathered before complete maturity, and by drying in that state turn blackish gray or brown. If left until quite ripe they lose some of their pungency and gradually fall off.

The berries after drying are spherical, about one-fifth inch in diameter, wrinkled on the surface, indistinctly pointed below by the remains of a very short pedicel, and

* London, McMillan & Co., 1879.

crowned still more indistinctly by the three or four lobed stigma. The thin pericarp tightly encloses a single seed, the embryo of which, in consequence of premature gathering, is undeveloped and merely replaced by a cavity situated below the apex. The seed itself contains within the thin red-brown testa a shining albumen, gray and horny without and mealy within. The pungent taste and peculiar smell of pepper are familiar to all.

The transverse section of the grain of black pepper exhibits a soft yellowish epidermis, covering the outer pericarp. This is formed of a closely-packed yellow layer of large, mostly radially arranged, thick-walled cells, each containing in its small cavity a mass of dark brown resin. The middle layer of the pericarp consists of soft tangentially extended parenchyme, containing an abundance of extremely small starch granules and drops of oil. The shrinking of this loose middle layer is the chief cause of the deep wrinkles on the surface of the berry. The next inner layer of the pericarp exhibits towards its circumference tangentially arranged, soft parenchyme, the cells of which possess either spiral striation or spiral fibers, but towards the interior loose parenchyme free from starch and containing very large oil cells. The testa is formed in the first place of a row of small yellow thick-walled cells. Next to them follows the true testa, as a dense, dark brown layer of lignified cells, the individual outlines of which are indistinguishable. The albumen of the seeds consists of angular, radially arranged, large-celled parenchyme. Most of its cells are colorless and loaded with starch, others contain a soft yellow amorphous mass. If thin slices are kept under glycerine for some time, these masses are slowly transformed into needle-shaped crystals of piperin.

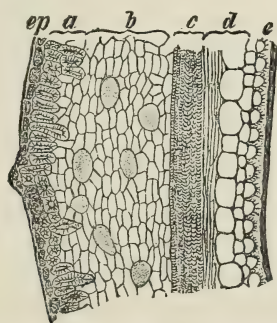


FIG. 11. Pepper husk, cross-section. *ep*, epidermis; *a*, stone cells; *b*, parenchyme with oil cells; *c*, spiral vessels; *d*, inner parenchyme; *e*, inner layer of stone cells. (After Schimper.)

Of the structure described so well in the preceding lines, of a portion of which a diagrammatic illustration is given in Fig. 11, after Schimper, only parts are readily found in the powdered pepper of the shops. The angular cells of the interior of the seed are of course the most prominent, and when once seen their characteristic form and contents are easily recognized again. The structure of the outer coats is made out with more difficulty. It is well, before attempting to do so on a ground pepper, to soften some whole black and white pepper-corns in glycerine and cut sections from various parts of the exterior of the berry. Taking the white pepper first it will not be found difficult in such sections mounted in glycerine to pick out three layers of different cells compos-

ing the outer coat of the corn, beside the angular large cells of the interior, which are filled with starch and piperine, the latter being yellow in color. The first of these layers and outer one is made up of colorless large loosely-arranged cells, with some fibers, more compact toward the exterior than the interior of the layer and carrying globules of oil. This layer makes up the principal part of the husk of white pepper. The second layer is a part of what Flückiger calls the testa, and consists of small yellow cells, thick walled and closely appressed. Next the third layer and second portion of the testa consists of lignified brown cells, which in their transverse appearance resemble some of the cells of mustard hulls, the individuality not being made out easily owing to the thickness of the walls. Having become thoroughly familiar with these appearances the white ground pepper should be examined and will be found to differ in the way in which these coats are presented. They can be recognized, however, and must be studied until thoroughly understood. The presence of the least portion of adulterant is then readily detected.

The black pepper is not as simple in its arrangement as the white. The maturity of the latter gives its structure more distinctness, while in the black the more or less shrunken character of the berry renders the recognition of the various tissues difficult. In a section from the exterior of a softened black pepper the interior coats, after what has been learned with the white, will be quickly recognized, but will be found to not be as plainly developed. The coats of the outer pericarp, which in the white pepper were wanting, will be found to be dark-colored, shrunken, and confused, so that it will require much study to discover the forms of cell which Flückiger describes. It will be found easier, perhaps, in the ground black pepper. There the structures already recognized in the ground white pepper will be seen and in addition dark-brown particles, portions of the outer coats. Careful examination of different particles will detect some which consist of the elongated vertical exterior cells, containing resin, while others are the shrunken parenchyma cells of the second layer, whose structure is indistinct.

Flückiger calls the first layer yellow, which hardly seems correct, as the appearance is nearly black. It is unnecessary, however, to attempt a minute study of these cells, as one is only required to be able to recognize their appearance and in addition to know something of the relative proportion of ground pepper which they should form as they are added in excess as pepper dust, the waste hulls of pepper being often used as an adulterant. The colored portion of a ground black pepper it will be found divides itself into two classes, the dark particles which have just been mentioned and the deep reddish ones, which are made up of the testa of the seed and its adherent parenchyma. The two will be readily recognized and distinguished from adulterants by the investigator.

The differences in the appearance of the peppers from various sources is sufficiently marked to be readily noticed when samples of each are placed side by side, but otherwise it is almost impossible to identify them. The best method of judging their quality and the one in use in the trade is by weight. Malabar is considered the heaviest. Blyth gives the following figures:

100 peppercorns of—	Grams.
Penang	6.2496
Malabar	6.0536
Sumatra	5.1476
Treva	4.5736
Tellicherry	4.5076

Tellicherry is the Malabar or West Coast port, so that variations must occur, as would be natural, in different samples.

We have found some samples to weigh—

Serial No.	Source.	Weight per 100 grams.	Percentage of dust and dirt.
<i>Black.</i>			
4514.....	Unknown.....	5.9000	
4840.....	Unknown.....	5.4600	
4894.....	Acheen.....	4.525	2.5
4895.....	West Coast.....	5.085	4.3
4896.....	Singapore.....	4.870	8.3
<i>White.</i>			
4516.....	Unknown.....	5.1300	
4898.....	Singapore.....	4.9600	1.4

The white pepper is of course the cleaner.

ADULTERANTS.

The common adulterants to be found in peppers are said to be flours or starches of cereals and potatoes, sago, mustard husk, linseed and capsicum, pepper dust, sawdust, gypsum, and other odds and ends.

Of the adulterations of pepper Blyth enumerates many, among them the celebrated pepper dusts designated as "P. D.," "H. P. D.," and "W. P. D.," and known as pepper dust, composed of linseed cake; hot-pepper dust, composed of mustard husks chiefly, and white-pepper dust, composed of ground rice. In this country the use of ground corn or rice, mustard hulls, cocoanut shells, and other similar refuse is very common, but whether they have been derived from goods sold as H. P. D. and W. P. D. we were unable to ascertain. Other refuse is also frequently found in cheap peppers, but sand does not seem to be as often added here as abroad. We have found the white peppers much freer from adulteration than the black. It is learned from Dr. Ellis, of Toronto, that roasted cocoanut shells are in common use now as an adulterant of all spices, and are of course easily introduced into peppers. They have not been found in the specimens which we have examined. Pepper husks,

mustard hulls, yellow corn, cracker dust, charcoal, and mineral matter have been detected by their characteristic appearances. The presence of pepper husks and charcoal is generally known by the immensely increased proportion of black particles in the field, as appears in Fig. 43, Plate XVII. A careful sorting of the coarser particles under a low power of the dissecting microscope and selection, for example, of the shining white grains of rice or yellow particles of corn from the more oily pepper cells, enables us to examine the starches separately with plain and polarized light. The appearance of the true pepper powder and one in which rice starch is present is given in Plate XXII, Figs. 43 and 44. Pepper starch is so much smaller than any other which we meet with, that it is not easily confused with it. Fig. 65, Plate XXVIII, gives an idea of its size as compared with others. With the dissecting microscope it is also convenient to go over the pepper powder and pick out any other class of suspicious particles for examination with higher power. With a little practice it soon becomes easy to tell an adulterated specimen even with a cursory examination with a lower power or a hand lens. In this way, in our experience, one or two foreign substances have been found which have not been identified, but which were evidently parts of the husks or coverings of some seed or fruit. The shell of the coconut, as has been said, has not been met with as an adulterant, but it is not at all difficult to identify, as its structure, with its innumerable stone cells and fibrous tissue, is very characteristic after treatment with Schulze's reagent.

The microscopic examination thus gives very certain indications of the quality of peppers, although frequently the eye without aid will detect a fictitious appearance. The chemical examination, on the other hand, as will be shown, serves as a reliable means of confirmation in many instances, and gives evidence of the quantity as well as kind of the adulteration. A considerable amount of information has been accumulated in regard to the proximate composition of peppers. Flücker quotes authorities for the statement that black pepper contains from 1.6 to 2.2 per cent. of volatile oil, of the character of a terpene and optically active; also a nitrogenous substance known as piperine to the extent of 2 to 8 per cent. Husemann and Hilger* give reference to papers by several investigators (see bibliography), and describes piperine as soluble in alcohol, and less so in ether. This substance, which is characteristic, will be found, therefore, in the ether extract of the spice, together with the volatile oil.

Blyth has made an examination of several peppers, and gives results which are useful for reference.

* Die Pflanzenstoffe, B. 2, S. 486.

Analysis of ash of Tellicherry pepper.

	Per cent.
Potash	24.38
Soda	3.23
Magnesia	13.00
Lime	11.60
Iron30
Phosphoric acid	8.47
Sulphuric acid	9.61
Chlorine	7.57
Carbonic acid	14.00
Sand	6.53

The sand, he finds, the most variable constituent, but never above 9 per cent. of the ash except in cases of willful adulteration. Phosphoric acid averages 8.5 per cent. of the ash, which is considered characteristic. He has also identified nitrates in peppers, finding in—

	Per cent.
Penang04470
Malabar03858
Tellicherry08860
Sumatra06560
Trang11870

The average proximate composition he finds to be—

	Per cent.
Volatile oil	1.04
Acrid resin	1.77
Piperine	5.17
Substances soluble in water, gum, starch, and other matters subtracting ash	14.74
Substances insoluble in alcohol and water	67.75
Water	9.53
	100.00

Following are some analyses made by Blyth in 1876:

	Hygroscopic moisture.	Piperine in pepper, dried at 100°.	Resin in pepper, dried at 100°.	Aqueous extract in pep- per, dried at 100°.	Ash in pepper, dried at 100°.	
					Soluble in water.	Total.
Penang	9.53	5.57	2.08	18.33	2.21	4.18
Tellicherry	12.90	4.68	1.70	16.50	3.38	5.77
Sumatra	16.10	4.70	1.74	17.59	2.62	4.31
Malabar	10.54	4.63	1.74	20.37	3.45	5.19
Trang	11.66	4.60	1.70	18.17	2.53	4.77
White pepper (com.)	10.30	5.60	2.0556	1.12
Long pepper	1.80	.80	16.82	4.47	8.30

The process for the estimation of piperine approved by Blyth is extraction with petroleum ether and treatment of the extract with sodic hydrate to remove the resin. Subsequent investigations by Lenz condemn this method, however.

More recently Rottger* has investigated the pepper-corn with especial consideration of the question of detection of adulteration, and decided that the examination of commercial peppers should be directed to determinations of the inorganic constituents, the percentage of water, and the microscope, with determinations of the soluble and insoluble ash and piperine in certain instances. He decides that the alcohol and ether extracts are of no value, quoting great variations in his results and those of others. He found—

	For ether extract, indirectly determined.	For alcohol extract of 90 per cent.
	<i>Per cent.</i>	<i>Per cent.</i>
White pepper.....	8.0 to 10.7	10.0 to 11.8
Black pepper	7.9 12.1	12.3 16.7

Borgman, Wolff, and Biechelet likewise have determined the amount of alcohol extract, but have neglected the ether extract. It seems difficult to understand why the alcohol extract should be selected, as the ether is much simpler, and refers almost directly to the amount of volatile oil and piperine which the spice contains, without so much of the indefinite resin and sugar which the alcohol extracts. The determination of water was made as follows by Rottger: The powdered pepper was placed for three hours over sulphuric acid, and a portion then weighed out and dried at 100° C. for one and a half hours, then for a quarter of an hour at a time until it begins to gain weight. He found the variations to be between 12.6 and 14.7 for black pepper, and 12.9 and 14.5 for white pepper.

This striking agreement, he considers, makes this determination of value. It is open to serious criticism and is of little value, in our view, for the following reason: By consulting our investigations on the hygroscopic character of organic matter in a state of fine division,† it will be seen that allowing peppers to remain over sulphuric acid for three hours would not accomplish the end desired, at least in our climate, viz, reducing the moisture in all to a constant figure, and in addition Rottger takes no account of the volatile oil lost at 100°. In our analyses we find, in fact, much less water than he does, as is usual in all organic material in our drier climate, and no greater variation among the peppers than the adulterated specimens. Practical tests of the method also proved unsatisfactory.

* Ber. über d. 4 Ver. Bayrisch. Vertreter der angew Chemie, 97-102.

† Vide Bibliography.

‡ Bull. No. 4, Div. of Chem., Department of Agriculture.

The per cent. of ash Rottger finds for black pepper to be between 3.4 and 5.1 per cent., white pepper between .8 and 2.9 per cent., with an exception in the case of Lampung pepper, which has 6.4 per cent. He concludes, with other investigators, that 6 is the highest allowable figure for ash in black pepper, with anything above 5 as suspicious. Three per cent. is, in the same way, the highest allowable figure for white pepper.

Of the composition of the ash he says:

The facts point to the conclusion that an exhaustive investigation of the mineral constituents in many cases may be of benefit in forming an opinion of the quality and purity of peppers. *

He found the following extremes :

	Black.	White.
Fe ₂ O ₃	2.2	
Mn ₂ O ₃89	
K ₂ O	27.4 to 34.7	5.1 to 7.1
Cl	5.6 8.7	.5 .9
SiO ₂	1.5 6.3	1.0 2.6
P ₂ O ₅ , water sol.11 .91	
P ₂ O ₅ , insol.	8.2 12.5	10.8 30.7

He gives the following detailed analyses :

Percentage composition of the pepper ash.

	Black pepper.		White pepper.	
	Unknown origin.	Malabar, 1883.	Unknown origin.	Singapore.
SiO ₂	6.36 to 1.61	1.54	2.62	1.46
HCl	5.59 6.83	8.71	.58	.90
SO ₃	4.03 4.05	4.00	3.24	3.75
CO ₂	17.28 20.10	19.17	11.90	10.01
P ₂ O ₅	11.10 9.46	11.06	29.34	30.75
K ₂ O	32.49 34.72	27.89	5.10	7.15
Na ₂ O	1.55 4.77	5.50	.74	.84
CaO	16.07 13.55	15.02	35.12	31.05
MgO	3.31 4.46	7.56	9.54	11.64
P ₂ O ₃	2.16 .99	.85	2.22	1.86
Mn ₂ O ₃81	.18	.89	.21

Rottger then discusses the determination of the piperine, and allows that for simplicity and accuracy drying the powder with milk of lime and extraction with ether is to be preferred. When the ether is pure, dry, and free from alcohol we find lime is of little use, or rather a complication, and that for purposes of detecting adulteration the ether extract alone furnishes all the information desired.

Ienz† has determined the amount of sugar produced by inverting the starch in fourteen peppers, and in the common adulterants, with acid. He found all the samples gave an equivalent of more than 50 per cent.

* Halenke has more recently discussed this subject: vide bibliography, Appendix A.

† Zeit. Anal. Chem., 1884, **23**, 501.

of sugar on the ash free organic matter of the pepper, while all the adulterants gave less than 30 per cent., with the exception of those which are starchy, as flours and meals.

Rottger repeated this determination, showing that some other substances besides starch were inverted by the acid, and obtained the following results :

Black pepper sugar equivalent.....	57.2 to 60.3
White pepper sugar equivalent	59.6 to 74.4

The Lampong pepper gave only 41.70. Such great variations he considers fatal to the method as a means of detecting adulterations. His conclusions seem not entirely justified, as a review of Lenz's figures will show.

Lenz in his paper affirms that any extract determination is useless, as with various adulterants the results may be very close to actual pepper. Petroleum ether he shows to be unreliable, the amount of extract depending on the kind of extraction apparatus used. There certainly should be no difficulty of purely manipulative detail of this description, for if sufficient time is given the solvent will work the same under all conditions, but not less than twenty hours' continuous extraction should be allowed, and it is not fair to generalize on an analysis where the extraction was continued less than that time, the poor results being due only to a probable faulty manner of manipulation, as we have met with no such trouble.

Lenz's conclusions seem hardly just, and while there may be cases where adulterants would not be detected by an extract determination, in the majority it is a great assistance. Lenz also refers to the method of separation of the powdered particles of pepper adulterants by means of liquids of certain specific gravities, and pronounces it usable. He prefers the treatment with iodine solution and the selection of the particles not blued for examination under the microscope as adulterants. We have found it perfectly simple and desirable to use the method of separation with sieves, and to examine the coarse powder, with or without treatment, under the microscope, when, with a little experience, it is easy to distinguish and recognize the source of particles not pepper after a careful examination with high powers.

He also casts doubt upon the value of an ash determination, and owing to the great variations which he finds quoted as possible, recommends calculating all results on an ash free basis. He, however, admits that a carefully and properly cleaned pepper-corn will not usually contain more than 6 per cent., and his strictures, therefore, do not seem to be consistent. He places reliance only on the process of his own invention, determinations of sugar corresponding to the starch and other invertible substances, with supplementary determinations of water and ash and microscopic examination. His results were as follows :

List of equivalent percentage of reducing sugars in ash free organic substance of peppers and adulterants.

No.	Name.	Ash.	Dry residue.	Ash free dry residue.	Sugar equivalent in sample.	Sugar equivalent in ash free dry sample.	Remarks.
1	Black Batavia pepper	3.85	87.68	83.83	43.8	52.3	Inverted directly.
2	Long pepper (2)	8.68	88.77	80.12	44.2	55.2	Inverted after extraction with water.
3	Black Singapore	3.62	86.88	83.26	43.9	52.8	Inverted directly.
4	Do.....	3.62	86.88	83.26	45.0	54.1	Inverted after extraction with alcohol.
5	Do	3.62	86.88	83.26	44.1	53.0	After water.
6	White pepper99	87.59	86.60	51.8	59.9	Do.
7	Palm-cake meal (1) ..	3.71	89.76	86.05	22.7	26.4	After water and alcohol.
8	Do	3.71	89.76	86.05	19.1	22.2	After water.
9	Do	3.71	89.76	86.05	22.7	26.4	Direct.
10	Palm-cake meal (2)...	3.65	89.35	85.70	{ 22.4	26.1	{ Do.
11	Do	3.65	89.35	85.70	{ 22.5	26.2	{
12	Palm-cake meal (3) ..	3.54	89.88	86.34	19.7	23.0	After water.
13	Palm-cake, pure	1.89	93.44	91.55	19.4	22.5	Do.
14	Pepper husks.....	15.61	89.50	73.89	11.5	15.6	After ether and alcohol.
15	Pepper husks picked out of commercial pepper.	9.21	88.64	79.43	13.0	16.4	After water.
16	Commercial pepper husks.	20.29	88.42	68.13	11.5	16.9	Do.
17	Commercial pepper powder containing palm-cake meal.	5.15	88.70	83.55	35.9	43.0	Do.
18	Pepper (3), with 28 per cent. of palm-cake meal (3).	3.59	87.88	84.29	36.0	42.7	Do.
19	Same with 42.4 per cent.	3.58	88.38	84.80	33.7	39.7	Do.
20	Walnut shells	1.04	89.34	88.30	17.7	20.0	Do.
21	Buckwheat meal.....	2.10	86.58	84.48	56.1	66.4	Direct.
22	Dried and roasted bread.	1.15	100.00	98.85	86.3	Do.
23	Do.....	1.15	100.00	98.85	62.6	After water.

Lenz's method of inverting was this : 3 to 4 grams of the substance to be examined was treated for three to four hours in a flask with 250 c.c. of water, being repeatedly shaken. It was then filtered off, washed with water, and the moist powder washed back into the flask, and to 200 c.c. of water 25 c.c. of 25 per cent. hydrochloric acid added. The flask was provided with a cork and tube one meter long and placed in a bath of boiling water and kept there three hours, with shaking. It is then made up on cooling to 500 c.c., after careful neutralization with soda. This liquid was then titrated with Fehling solution. When palm-cake was present it was found necessary to add a little zinc chloride to clear the solution. Most of the surrogates for pepper used in Germany Lenz found to be free from starch, so that this method would seem to be of wide applicability. He remarks, however, that other substances besides starch may be inverted, and for this reason it is necessary to adhere closely to one method of working. We shall from our own experience and that of others learn more in regard to the capabilities and usefulness of this method.

Haslinger and Moslinger* have also published some observations on pepper and its adulteration which are of interest. They show that in Germany the addition of grains of paradise, the seed of *Amomum Melegueta*, is a common practice whose detection can be accomplished most readily with the microscope, after a study of the structure of the grains of paradise, which is very characteristic. The starch cells are larger than in pepper, 3 to 6 times as long as hard, but the starch itself is scarcely distinguishable. The cells are arranged in parallel lines forming bundles pointed at the end. The cells remain white when treated with hydrochloric acid, while those of pepper are yellow.

These writers also looked into the ash of commercial samples of pepper and found from 4.1 to 27.0 per cent. of inorganic substance insoluble in hydrochloric acid where in peppers ground by themselves only .3 and .8 per cent. occurred. They also found pepper siftings, pepper husks, and other refuse in use as adulterants and followed up Lenz's suggestions as to determination of reducing sugar produced from the samples by acid, by examining pure pepper corns ground by themselves, and also pepper husks, and extending the determinations to cellulose also. The "dextrose," as they denominate it, was determined according to Allihn, the "cellulose" according to Henberger. The results were as follows :

	Dextrose.	Cellulose.
Black pepper, pure.....	56.0	15.6
Pepper husks, pure.....	16.4	45.0
Four samples for investigation :		
I.....	46.4	23.4
II.....	40.8	28.1
III.....	44.9	23.3
IV.....	41.5	26.8
Pepper siftings.....	21.6	37.4

From these figures they give the following formulæ for calculating adulterations where :

x =per cent. of pure pepper.

y =per cent. of hulls.

s =per cent. of dextrose or cellulose in sample.

a =per cent. of dextrose in pure pepper.

b =per cent. of dextrose in husks.

For the dextrose figures :

$$x = \frac{100s - 1640}{39.6}$$

For the cellulose figures :

$$x = \frac{4500 - 100s}{29.35}$$

* Ber. 4. Ver. Bay. Verterter ang. Chemie, 104-110.

Calculated on this basis the four samples were found to consist of:

	Pure pepper husks and pepper.	
	On the ground of dextrose det.	On ground of cellulose det.
I	75.8 and 24.2	73.5 and 26.5
II	61.6 38.4	37.5 42.5
III	72.1 27.9	74.0 26.0
IV	63.3 36.7	65.4 34.6

These figures show a fair agreement, and, in the opinion of the authors, entitle the method to consideration. They condemn, however, severely placing any reliance on determinations of the alcohol extract.

Weigmann* has also examined a number of pure peppers, and obtained results which do not harmonize with those of Lenz. He finds:

	White.	Black.
Ash	1.0 to 3.0	3.0 to 7.0
Starch (Lenz method) ..	55.0 63.0	32.0 44.0
Fiber	5.0 6.0	12.0 15.0
Water	12.0 to 15.0	

Our experience with the inversion of starch by acids is such as to make it seem very probable that, without the necessary attention to details, it would be very possible to obtain such results as those last given from lack of complete conversion of starch to reducing sugar or the inversion of other substances, and in the discussion of our analyses this will be referred to.

In the second report† of the Laboratoire Municipal de Paris the subject of pepper and its adulteration as practiced in France is discussed. There whole peppers are made artificially from plaster, gum, and a little pepper, and the ground article with the most diverse substances, such as hemp-seed cake, colza, rape and beechnut cakes, starches, residues from the manufacture of potato starch, mineral matter, sweeping of spice warehouses sold as pepper dust, and so forth. The residue from the manufacture of potato starch has, after fermentation, a pungent taste which makes it a desirable adulterant, but the most common one is the powdered kernel of the olive, which is yellowish white in color and possesses all the outward characteristics of white pepper and gives practically the same amount of ash. To give this powder the proper taste and pungency there was added Cayenne, powdered bay leaves, and dried and powdered orange skins. The mixture is recognized without difficulty by chemical and microscopic examination, the latter of which gives an absolute proof of the character of the substance.

It is recommended to separate the olive kernels from pepper by the use of a mixture of glycerine and water of a Sp. Gr. of 1.173 at 10° C., in which the former sink. After decantation they can be examined

* Rep. anal. Chem. 6, 399-40.

† Documents sur les falsifications des Matieres alimentaires, Paris, 1885, p. 688-695.

microscopically. The structure is that characteristic of the hard thickened sclerenchyma or stone cells, polyhedral and elongated in shape with thickened walls and little filiform canals. They are most readily made out with polarized light, toward which they are optically active, while the remaining particles of pepper or pepper husks are not seen in the dark field, being inactive.

Of this adulterant H. Rabourdin has made some investigations, publishing his results in an interesting paper,* giving a general account of the various peppers of commerce and the use of olive stones as an adulterant, as well as other materials already mentioned. He finds no difficulty in recognizing the falsification owing to the presence of the numerous sclerenchyma or stone cells, which are distinguished readily with polarized light, but the quantitative determination is more difficult. This, however, he affirms, can be accomplished by determining the residue left on boiling the suspected sample for one hour with 1 per cent. sulphuric acid after thorough washing with water. In the course of the operation the presence of the olive stones is apparent from their clinging in the form of a reddish powder to the walls of the flask or beaker in which the digestion has taken place, and separating more or less from the pepper hulls on account of their different specific gravity.

For pure peppers the following percentage of residue was found:

	Per cent.	Mean.
Tellicherry	30.3 to 27.8	29.4
Alepy	31.5 31.8	31.7
Saigon	28.7 29.2	29.0
Singapore	33.2 34.0	33.7
Penang	33.8 34.2	34.0
Sumatra	33.0
Java	37.5 37.7	37.6
White, pure	17.5	17.5
Powdered, pure	29.2 29.0
Do	30.5 35.5

On the other hand olive stones were found to contain:

	Per cent.	Mean.
Coarse gray	74.2 to 75.0	74.5
Fine gray	74.2 74.6	74.4
Coarse white	74.5
Fine white	74.5
Pepper dust	65.5

From these figures the following factors are derived:

	Per cent.
White pepper	17.5
Malabar, Tellicherry, and Saigon	30.0
Alepy	32.0
Others known as light peppers	35.0
Olive stones	74.5
Pepper refuse	75.5

*J. de Pharm. et de Chimie, [5], 9, 289-297.

And if y is the per cent. of residue, a the per cent. in pure pepper, b the per cent. in olive stones, the amount of addition can be calculated from the following equations:

$$\begin{aligned}x + y &= 100 & ax + by &= p \\ y &= \frac{p - a}{b - a}\end{aligned}$$

where b can be replaced by 74.5 or 65.5 as the case may be.

Working in this way 10 ground peppers, guaranteed pure, were found to contain from 40 to 60 per cent. of olive stones, and these contained "P.D." to the extent of 14 to 20 per cent.

Control experiments with known mixtures seem to have been satisfactory.

The same author also extended his experiments to a comparison of the properties and separation of the cellulose derived from a mixture of pepper and olive stones, depending upon the difference in specific gravity, with results which can be found in his original paper.

The residues of starch factories always contain enough starch grains to make their identification easy.

Very recently Chas. Heisch* has given in the Analyst his experience in the examination of peppers, to see if there were any reliable mode of judging of the amount of adulterants in adulterated samples except by determinations of ash which give no indication of added organic matter. He also endeavored to find if it were possible to cleanse pepper corns so that the ash should be free from sand. Assisted by a large buyer and grinders of pepper he found that as the result of the grinder's experience no ground pepper should be sent to market which contains over 6 per cent. of total ash. In this respect they agree with the Bavarian chemists. He also placed some faith in the determination of starch and calculated, as did Lenz, his results on dry ash free organic matter.

The starch was estimated "by boiling the finely ground pepper for three hours with 10 per cent. hydrochloric acid, and taking the rotation in the resulting liquid." He endeavored to check his results by determining that there was insufficient "gum or other matters to affect the rotatory power" but neglects to see how much of the substances allied to cellulose are converted into optically active substances which would probably be important. Stating the results, however, as reducing sugar equivalent to pepper with Lenz, the error becomes of slight importance.

The determination of piperine was rough, and the results seemed of little service.

*Analyst, 2, 186-190, October, 1886.

The results were as follows:

No.	Name of sample.	Water.	Ash of dry pepper.					On ash and water free pepper.		
			Total.	Soluble in H ₂ O.	Soluble in HCl.	Insoluble.	Alkalinity as K ₂ O.	Starch.	Alcohol extract.	Piperine.
	Black Pepper:									
1	Acheen Penang	9.46	8.99	1.54	3.07	4.38	.72	48.53	12.26	6.04
2	Trang	9.22	8.85	1.60	3.83	3.42	.81	54.06	12.28	4.05
3	Singapore	14.36	5.41	2.07	2.52	.82	.91	56.24	12.41	7.14
4	Tellicherry	13.76	5.28	3.34	1.90	.04	1.41	56.67	12.67	6.88
5	Penang	12.98	6.45	3.10	2.44	.91	1.19	51.06	16.20	9.38
6	Tellicherry (brushed)	13.01	6.41	2.38	2.84	1.20	1.57	55.87	13.62	7.86
7	Gray light dusty Singapore	13.94	5.39	2.48	2.18	.73	1.09	54.93	11.62	6.30
8	Good B, Singapore	14.10	4.35	2.48	1.51	.36	1.14	54.54	10.47	6.06
	White Pepper:									
9	Penang	15.86	3.78	0.62	2.80	.36	.22	77.68	9.73	5.54
10	Singapore	17.32	1.28	.22	0.85	.22	.00	76.35	9.49	6.14
11	Siam	13.67	1.81	.25	.92	.69	.11	76.27	9.23	5.13
	As ground for market:									
12	Fine white	13.90	1.58	.16	.90	.52	.00	75.31	10.60	4.51
13	Finest	14.13	2.18	.50	1.50	.17	.11	84.69	9.53	4.70
14	I super	14.40	1.41	.37	1.03	.00	.11	85.26	9.63	4.50
	Long pepper:									
15	No. 1, H	12.15	13.48	2.28	5.52	5.68	.53	58.98	8.29	1.71
16	No. 2, T	14.93	11.98	2.37	5.83	3.69	.82	46.16	8.52	1.70
17	Black pepper husks	12.37	11.90	2.12	6.37	3.41	.48	41.71	13.81	4.84
18	Do., with same whole	12.60	9.04	3.00	4.12	1.92	.02	47.36	13.07	4.10
19	Sifting before grinding	7.96	51.39	1.02	6.47	43.90	.80	30.66	7.52	1.15
20	Black pepper, for sale of which fine was inflicted	11.12	14.70	2.02	4.07	8.61	.80	35.85	11.57	2.02
21	Poivrette used to mix with pepper	8.52	3.85	.96	1.05	1.84	0.21	0.00	2.31
22	10 per cent. poivrette, 90 per cent. No. 4	13.34	5.04	2.88	1.78	.38	1.14	49.98
23	30 per cent. rice, 70 per cent. No. 4	12.76	3.10	1.68	1.39	.03	.89	88.21

Of the above samples Nos. 1 to 5 and 7 to 8 were black pepper corns ground just as imported. The uniformity in the starch in all these lead Heisch to believe that any result under 50 per cent. should be regarded as suspicious, it being very easy to detect the addition of foreign starch, such as rice. Of the white peppers the first three are white pepper corns ground as imported, the next three black corns decorticated in England and then ground.

Of the long pepper Heisch says the starch is double the size of the ordinary and much more angular and like rice. Care must be taken therefore not to confound them. The poivrette is made from olive stones.

The figures are of interest and would seem to confirm the work of Lenz.

Prof. J. Campbell Brown has devoted much time to the subject of pepper and has very recently called attention in England to an adulterant which first made its appearance in Liverpool last summer and since then has been often met with. It is known as pepperette or poivrette, and proves to be the same adulterant so often mentioned in France, olive stones. It resembled walnut shells and almond shells somewhat, but olive stones more so.

Brown gives the following analyses and says:*

	Ash.	Matters soluble by boiling in diluted acid.	Albumi- nous and other mat- ters soluble in alkali.	Woody fiber, insol- uble in acid and alkali.	Starch.
White pepperette	1.33	38.32	14.08	48.48	None.
Black pepperette	2.47	34.55	17.66	47.69	None.
Ground almond shells	2.05	23.53	24.79	51.68	None.
Ground olive stones.....	1.61	39.08	15.04	45.38	None.

The stones of olives, imported in pickle for table use, gave 3.68 per cent. of ash, but well-washed olive-stones, thoroughly burnt to a white ash, gave under 2 per cent. of ash-like poivrete. "White poivrete" is therefore cleaned very pale, and perhaps partly bleached olive-stones or precisely similar tissue; black poivrete is the same, mixed with a little black husk. It is to be noted that although it contains no starch, yet it yields some sugar to Fehling's solution, after being boiled for some time with dilute hydrochloric acid. The quantity depends on the length of time and strength of acid, but may be stated approximately about 10 per cent. It is important to bear this fact in mind when making a full chemical analysis of pepper containing poivrete. After removing from such a mixture the matters soluble by boiling in dilute caustic alkali, the woody fiber which remains has a yellow color. It consists of the poivrete and some of the cells of pepper husk and one of the subcortical layers of the pepper berry. The pepper cells are made lighter and the poivrete cells darker by the alkali, so that the two are more nearly of a similar yellow color after treatment with alkali. This renders it more difficult to distinguish such of the cells as have somewhat similar markings, but it enables us to distinguish more clearly as poivrete the many torn particles which have no definite form or markings. The final examination of the complete cells is better made with good daylight rather than with artificial light, and in a portion which has been treated with water only.

The pepper cells are mostly different in shape and are colored, and have generally a dark substance in the interior. They are not numerous, but the quantity varies in commercial samples, owing to the modern practice of decorticating the pepper berry to every different extent possible, and mixing the various portions so obtained, including husks, in every variety of proportion with each other or with ordinary pepper. Each individual analyst must make himself familiar with both kinds of cells, as no description can convey an adequate idea of either. In order to form a judgment regarding the proportions of the different chemical constituents of commercial samples, we require to know the chemical composition of the different layers of the pepper-corn, and I hope soon to communicate to the society some figures bearing on this point, as well as to notice some other substances used in the sophistication of pepper.

It is interesting to note that the exemption, mentioned in section 8 of the sale of food and drugs act, in the case of a label being affixed to the article sold intimating that the same is a mixture, does not apply in the case of poivrete, the admixture being made manifestly for the purpose of fraudulently increasing the weight and bulk.

In a subsequent note Brown† warns analysts not to confuse an excessive amount of cortical cells of the pepper husk for poivrete, as they are somewhat similar. This, however, would certainly not occur if authentic samples of pepper and olive-stones were used for comparison.

Brown also contributes a paper to the Analyst§ on the use of "long pepper," *Chavica Roxburghii*, as an admixture to the ordinary ground

* Analyst, **12**, 23-25.

† Analyst, **12**, 47-48.

§ Analyst, **12**, 67-70.

article, showing that it should be discouraged, from the fact that it brings with it a large amount of dirt and mineral matter, and has a disagreeable offensive odor developed by warmth. Brown says:

It is now time that all should take up a decided position in regard to this form of adulteration. Long pepper is the fruit of *Chavica Roxburghii*,* and does not consist merely of the berries analogous to the pepper-corns of the true pepper-plant; it bears much the same relation to them that wild grass-seed would bear to oatmeal. It consists of the small berries with the husks and indurated coverings hardened together and to the central woody stem, much in the same way that in pines the seed and coverings are all hardened into one cone. Long pepper is for the most part derived from wild plants of *Chavica Roxburghii*, which grow by the sides of the water-courses in India. Consequently it always brings with it a mass of dirt, picked up from the soil of the banks whereon it grows, embedded in the crevices and irregularities of the fruit, which dirt the native collector takes care not to lessen, but rather to increase, seeing that he is paid by weight for what he brings down to the merchants.

In commerce we find accordingly that it has always from 3 to 7 per cent. of insoluble sand and clay, in addition to the proper ash of the fruit. And it is difficult, if not impossible, to clean long pepper before grinding in the way that true pepper can easily be cleaned; it can with difficulty be cleaned by hand.

The ash contains a very large proportion of salts insoluble in hydrochloric acid. When ground, the hard husk and woody center, as well as the dirt, are necessarily ground along with the minute berries. The ground long pepper contains not only sand, but more woody fibre than ground genuine pepper of the corresponding shade, although not so much total cellulose as the most husky black pepper. It has the composition shown by Mr. Heisch in his paper. I can confirm his results by the following:

Analyses of long pepper carefully cleaned by hand.

	Total a	Sand and ash insoluble in HCl.	Total matter soluble in 10 per cent. HCl.	Starch and matters convertible into sugar.	Albuminous matter soluble in alkali.	Cellulose.	Extracted by alcohol.	Extracted by ether.	Total nitrogen.
1	8.91	1.2	67.83	44.04	15.47	15.70	7.7	5.5	2.1
2	8.98	1.1	68.31	49.34	17.42	10.50	7.6	4.9	2.0
3	9.61	1.5	65.91	44.61	15.51	10.73	10.5	8.6	2.3

Although the cost of long pepper is at present nearly as high as some very inferior varieties of black pepper, yet the price is generally decidedly lower; even now long pepper is much cheaper than the pepper with which it has been sometimes mixed of late, and its use affords a handsome illegitimate profit, to the detriment both of the grocer and his customer. Long pepper has been, and is, legitimately used for pickles, but it is not known, nor has it been recognized by the trade, as ground long pepper; and all the respectable grocers, and others of whom I have inquired, say decidedly that they would not buy, nor retain, if received, any ground pepper which they knew or suspected to contain an admixture of long pepper. In fact, it is no more right to give pepper containing long pepper in response to a request for simply "pepper" than it would be to give horse-chestnuts instead of Spanish chestnuts in response to a request for simply "chestnuts." It may, of course, be sold as ground long pepper without offense, but no one would buy it. Not only is long pepper a fraudulent admixture in ground pepper, but it is objectionable on the score of quality and flavor. Its disagreeable, offensive odor is developed by warmth. Any candid person can convince himself of the real cause of the objections which housekeepers and grocers

* Known also as *Piper officinarum* and *Piper Longum*.

alike have to ground long pepper if he will heat up a piece of cold meat between two plates and sprinkle some fresh long pepper on it; the smell and flavor are so offensive that he will feel obliged to reject the meat.

Much of that which one gets whole in shops is very old, and has lost much of its flavor and strength, so small a sale does it command.

The presence of long pepper in ground pepper may be determined by the following characters:

1. *Color*.—If any serious quantity of long pepper is ground in with the ordinary pepper it imparts some of its peculiar slaty color; but this is made much lighter by the now very common practice of sifting out much of the darker or husky portions of the long pepper before mixing it with the genuine pepper. Bleaching is also resorted to, but not hitherto very effectively.

2. The odor of the mixture when warmed is unmistakable by an educated olfactory sense, even if the quantity is comparatively moderate. Attempts are made to disguise the odor by bleaching, but this has not been successful. The ethereal extract also, and even the alcohol extract from which the solvent has been evaporated at a low temperature, yields, when warmed, the characteristic odor very plainly.

3. Long pepper introduces sand into the pepper with which it is mixed, often to a considerable amount. If the pepper is white, this has more importance than has hitherto been accorded to it; for white pepper does not contain, even as imported, 2½ per cent. of sand, and any white pepper containing so much sand must have had the sand improperly introduced, either by direct mixing of Calais sand or in some other way.

Long pepper from which the husk particles have been sifted out when added to white pepper invariably introduces its sand along with it, as well as some spent bleach, if attempts have been made to bleach it.

4. The woody matter in ground long pepper is always considerable, arising both from the smallness of the berries, compared with the hardened setting, and from the central woody tube. This may be detected either by chemical analysis or by the microscope, and some of it by the naked eye or a large hand lens.

If the sample is spread out in a smooth thin layer on strong paper, by means of an ivory paper-knife, pieces of fluffy woody fiber will be detected, especially if the smooth thin layer be tapped lightly from below. Those pieces come from the central part of the indurated catkin which cannot be completely ground fine as genuine pepper stalks are, and are very characteristic if carefully examined. Much of these are of course removed by the grinders' sieves, but enough finds its way through the meshes of the silk to be useful as a corroborative indication.

5. Particles of husks, if present, can be distinguished from genuine pepper husks.

6. A proportion of the starch granules of long pepper are of larger size, above .0002 inch, and of angular shape, very slightly smaller than rice granules, and more loosely aggregated in clusters or isolated.

Brown also calls attention to the statement of authorities that genuine pepper starch is round in form, and shows that this is not always the case. By reference to our illustration, Fig. 65, Plate XXVIII, it will be seen that he is correct. He has lately found* that Dhoura corn, a variety of sorghum, is being largely used in England as a diluent of pepper. The grain is well known in this country as Egyptian corn, and is a common crop in the South and Southwest, but has not been used here as an adulterant. Brown says:

I have met with it only about four times in pepper, but it probably occurs more frequently in other districts. It is known in England as great millet or Turkish

* Analyst, 12, 89-90.

millet, and is the grain (with an integument, but without the husk) of one of the cereal grasses, *Sorghum vulgare*.

It is a roundish or oval somewhat flattened grain, size from one-eighth to one-fifth of an inch in diameter, white in color and brittle, with a thin, smooth integument or testa, showing under a high microscopic power, on the inner surface, an aggregation of very small granules, which become blue by iodine. The body of the seed is very white, and consists mainly of roundish or irregular starch granules, varying in size from .0001 up to .0006 of an inch in diameter, and showing under polarized light a nearly right-angled cross; and of larger irregularly rounded granules of starch from .0005 up to .0013 of an inch in diameter, showing no cross, or only a very faint one, under polarized light.

Some of the first-named granules have a hilum and star in the centre, somewhat like bean starch. By boiling with caustic alkali the cellular membrane which binds the starch granules together is disclosed.

The influence of an admixture of sorghum with pepper upon analysis of the latter will be seen from the following analysis of sorghum grains:

Moisture 11 per cent.

Composition of the dried sample.

	I.	II.
Ash	1.31	1.69
Soluble in 10 per cent. hydrochloric acid	90.70	87.80
Starch	75.20	73.00
Albuminous matters soluble in caustic alkali	6.71	7.96
Cellulose	2.56	4.19
Alcoholic extract	10.36	7.96
Ethereal extract	10.10	7.30
Nitrogen	1.82	1.79

There would be no difficulty in detecting it.

Although as yet these substances do not seem to have reached us as far as the samples which we have examined show they must be carefully watched for.

In this country considerable has been published as to the adulterations of pepper, but little in regard to the manner of detecting them. We have already spoken of the large numbers of samples which have been examined in different years by the public analysts of Canada and smaller numbers by those of Massachusetts and New York. Reference to the reports of the department of inland revenue of the Dominion (supplements on adulteration of food) shows that wheat flour, husks, cayenne, coconut shells, and pepper dust, milling refuse, pea meal, and sand and clay are in very common use. In the United States the samples examined have apparently proved no better, for while in Canada in 1885 twenty-nine out of sixty samples were adulterated mostly from 10 to 20 per cent., but reaching 75 per cent.; in New York in 1882, out of forty-seven, thirty-three were adulterated, and in Massachusetts in 1884 Wood found one hundred and four in one hundred and ninety-nine impure, and in 1883 69 to 70 per cent. were bad.

We are thus supplied with considerable experience in the examination of peppers, the results of which furnish the basis of a scheme for general use. Thus in examining a sample I should propose to proceed as follows:

METHOD OF EXAMINING PEPPERS MICROSCOPICALLY.

As a preliminary the sieve examination, already mentioned, is of value, the coarser particles left upon a 40 or 60 mesh sieve frequently revealing the nature of the adulterant or the too large proportion of pepper husk. Afterwards it is well, with a good dissecting microscope and a power of 15 to 30, to sort over the ground pepper, and judge of the frequency of the occurrence of the coarse particles, which after a little experience there will be no difficulty in doing. The presence of sand or a notable excess of P. D. can also be detected and estimated in this simple way. Backgrounds of white and black, with reflected light and afterwards transmitted light, may be used in the manner so conveniently afforded by Zeiss's stand, made for this purpose.

A portion of the powdered pepper or the separated coarse particles should also be treated with chloral-hydrate solution for twenty-four hours, to render it more transparent for examination with higher powers, and in the mean time the coarse particles sieved from the powder may be examined under a one and a one-half inch objective, and then crushed and re-examined, using both plain and polarized light. In this way husky matter may be distinguished and foreign starches detected. Polarized light is then the means of bringing out more plainly the starches, the proportion of which iodine will reveal, making due allowance for the small granules of pepper starch and all optically active tissue, such as the bast fibers and sclerenchyma or stone cells, which are found in olive-stones and cocoa-nut shells.

The structure of the pepper itself, as has been explained, is so characteristic as not to be readily confused with foreign matter. In the chloral hydrate preparation, which should now be examined, much of this disappears as the starch is much swollen by this reagent. The husky matter present is rendered thereby so much clearer on the other hand that its identification and differentiation is made much easier, and it is here that the possibility of fixing the source of the adulterant will often lie.

Experience with a half dozen samples from a cheap grocery in comparison with a laboratory sample of pure pepper will soon teach one the best means of making out what has been briefly described.

It has also been found most valuable to digest about a gram of pepper with nitric acid, sp. gr. 1.1, and chlorate of potash for several hours, or until the color is bleached. It is then possible to distinguish the denser cellular structure more easily than in any other way, particularly the stone cells which make up the larger part of the cocoanut shells and ground olive stones, especially with polarized light, being careful not to confuse the stone cells of the pepper husk with those of olive stones or other adulterants. Charcoal at the same time remains unbleached.

To determine the merits and correctness of the various chemical processes and statements in regard to them previously referred to, a series

of pure whole peppers, direct from importers, and of the commercial ground article, have been collected. The results also reveal the extent and nature of the adulteration practiced in this part of the country. The specimens were of the origin described below.

Sources of specimens of pepper.

WHOLE PEPPERS.

No.	Kind.	Remarks.
4514	Whole black....	First quality, probably west coast.
4840	... do	West coast, direct from importer.
4894	... do	Aachen, direct from importer.
4895	... do	West coast, direct from importer.
4896	... do	Singapore, direct from importer.
4516	Whole white....	First quality, source doubtful.
4898	... Do.....	Singapore, direct from importers.
GROUND PEPPERS.		
4515	Black.....	First quality, grocers' guaranteed pure.
4523	... do	Ground in Washington.
28	... do	Ground in Baltimore.
33	... do	Do.
37	... do	Do.
43	... do	Ground in London, guaranteed
52	... do	Ground in Washington.
53	... do	Do.
4883	... do	Ground in Baltimore, low grade "Best."
4884	... do	Ground in Baltimore, low grade "Pure."
4524	White.....	Ground in Washington.
4544	... do	Ground in London, "Pure."
4555	... do	Ground in Washington.
4882	... do	Ground in Baltimore.

MECHANICAL AND MICROSCOPICAL EXAMINATION.

The weight of the whole peppers and the amount of dirt present, as they are imported, have been given already. In the ground condition they of course displayed the normal structure of the berry, as has been already described. No further reference is necessary, therefore, under this head. In the ground specimens the following peculiarities were noted.

4515. Sifting reveals the presence of pepper stems which should not be present, showing that the pepper was ground without cleaning and an undue proportion of husky matter, unbleached by nitric acid and chlorate.

4523. This specimen is very coarsely ground, a large proportion of husk remaining on the 40-mesh sieve, among which evidence of the presence of maize could be distinguished, and that P. D. in some form must be present.

4528. Sifting separated light chaffy and fibrous matter. The microscope detected yellow corn and its hulls.

4533. This proved of very bad quality, the siftings consisting of bran, roasted shells or charcoal, and corn. The microscope was confirmatory, and the presence of the roasted shells prevented bleaching with Schulze's reagent. This is evidently a P. D. pepper of the worst sort.

4537. Sifting and examination showed the presence of P. D. in some form and corn. It contains no roasted matter or charcoal.

4543. Proved to be quite pure and well ground, all the material passing a 40-mesh and nearly all a 60-mesh sieve. This is the only *pure ground* sample met with.

4552. Sifting shows the presence of a complicated collection of adulterants, husks of various origin, &c. Microscopic examination detected mustard hulls, corn, roasted shells, or charcoal not bleachable, and other foreign material not identified.

4553. Contains mustard hulls and branny matter, but no charcoal. *Bad.*

4883 and 4884. From the same mill in Baltimore were the worst specimens met with. They contained but little pepper and were made up of P. D., yellow corn, cracker dust, cayenne, charcoal, and other foreign matter.

4882. A white pepper from the same source was of the same origin, leaving out the black elements.

4524 and 4544. These white peppers were found to be pure, but the former not carefully decorticated.

4555. Contained foreign starchy matter and probably cayenne. Sifting revealed nothing abnormal.

These examples serve to show the variations which are met with and what the analyst may expect. It is always well, also, to be on one's guard for something new.

As a confirmation of the physical examination and a means of determining the amount of adulteration in the several cases determinations were made of the proximate composition:

Analysis of peppers.

[Whole black.]

Serial number.	Source.	Percent. of dust.	Weight of 100 grains.	Quality.	Water.	Ash.	Volatile oil.	Piperine and resin.	Alcohol extract.	Starch.	Undetermined.	Crude fiber.	Albuminoids.	Total.	Total N. x 6.25.	Total N.	Reducing sugar equivalent on dry ash free pepper.
4514	West Coast	Clean.....	5.900	Cleaned.....	8.91	4.04	.70	7.29	5.08	36.52	24.62	10.23	7.69	100	9.81	1.57	47.16
4840	do.	Clean.....	6.460	Retail.....	8.15	2.91	1.48	7.20	5.08	33.32	21.62	8.74	11.30	100	13.63	2.18	42.38
4894	Acheen	2.5.....	4.525	As imported.....	8.29	4.70	1.69	7.72	6.06	37.50	13.64	10.02	10.38	100	12.60	2.02	47.87
4895	West Coast	4.3.....	5.085	do.....	9.36	4.52	1.63	7.90	5.71	36.18	13.59	10.30	10.81	100	13.13	2.10	46.08
4896	Singapore	8.3.....	4.870	do.....	9.83	3.70	1.60	7.15	5.74	37.30	17.66	10.02	10.00	100	12.08	1.93	44.13

[Whole white.]

4516	West Coast	Clean.....	5.130	Retail.....	9.85	1.41	.57	7.24	2.57	40.61	23.28	7.73	9.31	100	11.48	1.83	50.86
4898	Singapore	1.4.....	4.960	Imported.....	10.60	1.34	1.26	7.76	2.57	43.10	19.55	4.20	9.62	100	11.90	1.90	54.38

[Commercial ground black.]

4515	Washington grocers	Adulterated.....	10.00	4.52	.69	6.50	31.86	21.53	15.90	9.00	100	10.88	1.74	39.80
4523	do.	do.....	11.83	4.38	1.04	6.59	21.56	23.49	10.55	20.56	100	21.88	3.50	28.52
4528	do.	do.....	12.30	6.54	.84	5.94	30.00	18.94	15.43	9.51	100	11.20	1.79	40.90
4533	do.	do.....	8.92	4.06	.92	3.99	30.83	21.52	20.48	9.28	100	10.33	1.65	39.38
4537	do.	do.....	11.15	7.85	.82	6.59	27.85	17.30	19.03	9.31	100	11.20	1.79	38.19
4543	do.	Good.....	10.70	5.85	.42	7.19	30.47	20.81	14.30	9.66	100	11.73	1.88	40.37
4552	do.	Adulterated.....	7.60	7.50	2.94	5.92	27.04	25.55	11.20	12.25	100	13.95	2.23	35.40
4553	do.	do.....	8.90	9.85	1.14	6.38	21.27	27.13	13.53	14.80	100	16.63	2.66	29.08
4883	Baltimore Mills	do.....	9.48	6.15	2.09	2.66	6.54	28.19	14.83	18.15	11.93	100	12.60	2.02	37.13
4884	do.	do.....	8.45	5.29	1.06	2.80	5.64	31.34	16.36	16.73	11.73	100	12.43	1.99	40.37

[Commercial ground white.]

4524	Baltimore Mills	Pure.....	11.56	1.16	.48	7.11	41.06	25.30	5.13	8.20	100	10.33	1.65	52.28
4544	do.	do.....	11.35	1.86	.55	7.45	48.20	14.35	4.63	12.52	100	13.80	2.20	59.81
4555	do.	do.....	8.95	1.35	1.25	7.22	50.30	16.55	5.98	8.40	100	10.50	1.68	65.73
4882	do.	Adulterated.....	11.12	2.13	.98	2.84	4.10	15.58	4.24	12.13	10.79	100	11.55	1.85	66.09

The analyses of the pure specimens of pepper show that in all the amount of water was between 8 and 10 per cent. This is of course variable with surrounding conditions. The ash in black peppers did not exceed 4.7 per cent., and in white 1.4. It is fair to believe that anything above 5 per cent. for black and 2 per cent. for white is suspicious, otherwise adulteration or dirt are certainly present.

The volatile oil varies in black pepper from 1.69 to .70, and in white 1.26 and .57 were found. This determination is not of great value as a means of detecting adulteration. Piperine and resin, however, furnish a most valuable check on the purity of the samples. In the whole berries ground by us both black and white contained from 7.90 to 7.24 of these substances, showing great constancy in amount, and on the addition of adulterants this is plainly affected. It serves, in my opinion, as well or better than determinations of pure piperine, the latter being difficult and involving loss, as has been shown by careful experiments made by Mr. Knorr under my direction. It has also proved impossible to make determinations of piperine by the combustion or Kjeldahl methods by application of Stützer's copper hydrate process, the per cent. of nitrogen being so small, 4.912 in piperine, as to make the error very large when converting the former to the latter, the necessary factor being 20.36. Rottger's objection to the value of the determination of the ether extract is not sustained in our experience, and it seems that he must have employed inferior ether, which is often the cause of serious error. Only the best Squibb's ether or its equivalent should be used for extractions.

The alcohol extract appears to be of no value.

The determination of starch or its equivalent in reducing sugars has been looked into with care and the conclusions arrived at are that the results are of value when carried out in the manner which has been described under the general head of methods of analysis. A preliminary extraction with alcohol and water is necessary to obtain results which are fairly constant. Determinations made in this way show that black peppers contain from 34 to 38 per cent. of starch, or 42 to 47 per cent. of substances of reducing sugar equivalent calculated on dry ash free substance. White peppers contain in the same way from 40 to 43 per cent. of starch, and give from 50 to 55 per cent. of reducing sugar equivalent on dry ash free substance. These figures are not as high as those given by Lenz, but are the result of careful work on pure samples, extending over a long time and involving much experience. It is believed that they must be nearly correct. In our opinion there is no advantage in calculating the results to a sugar equivalent unless excessive moisture or ash is found to be present.

The crude fiber in the black peppers, as determined by our methods, does not vary far from 10 per cent. One sample contained but 8.74, and of course in the white peppers the amount is much reduced, depending to a certain extent on the perfection of the decortication; 4 to

8 per cent. are probably fair limits. The determination is of comparative value, revealing the presence of foreign woody or fibrous matter.

The albuminoids do not vary widely, 10 per cent. being the average, with extremes in our experience of 7.69 and 11.50. The addition of nitrogenous seeds of course increases the amount, and of fibrous or woody matter diminishes it. As will be seen, therefore, the determination is a useful one. The variations, then, may be summarized as follows:

	Black.	White.
Water	8.0 to 11.0	8.0 to 11.0
Ash	2.75 to 5.0	1.0 to 2.0
Volatile oil50 to 1.75	.50 to 1.75
Piperine and resin	7.0 to 8.0	7.0 to 8.0
Starch	32.0 to 38.0	40.0 to 44.0
Crude fiber	8.0 to 11.0	4.11 to 8.0
Albuminoids	7.0 to 12.0	8.0 to 10.0

With these pure peppers the ground samples must be compared. In the table the determinations which are suspicious are printed in full-faced type. All but one of the black peppers are convicted of adulteration on the chemical evidence.

The first, 4515, has too little piperine and resin, too little starch, and too much fiber. It is apparent that some fibrous or woody diluent has been added, as appears from the microscopic examination to be true.

Without discussing each analysis in detail it may be pointed out that only three samples contained an excess of ash, as a rule being free from mineral adulterants, and the three cases are probably only dirty, so that the addition of minerals is not common in this country.

The piperine and resin were deficient in all the specimens except the one pure one, revealing at once sophistication.

Starch was deficient in all the specimens, in the pure specimen falling below the usual limits, which at the same time was more than usually husky, thus accounting for the difference, and in fact in all but two cases there was present more fiber in the peppers than we have considered normal, another indication of the presence of adulterants or dirt.

The albuminoids in two cases were present in excessive amount, revealing the presence of some foreign seed rich in nitrogen. In the other specimens the adulterants did not throw this determination far away from the normal.

In the white peppers the presence of an excessive amount of starch in 4555 makes it suspicious, and the great deficit in piperine and excess of starch and fiber show that 4882 is adulterated.

The value of the chemical determinations is thus plainly illustrated. Perhaps with no one of the substances which are considered in this report are the indications more certain. It would be possible, too, in an indirect way, by means of proportions such as have been already mentioned, to calculate with an approximation to accuracy the extent of the

adulteration. It is fortunate that in a material which is probably more frequently adulterated than any other the presence of foreign matter is so easily detected.

CAYENNE.

Cayenne or red pepper is the powdered pod of several species of *Cap-sicum*, a genus of the family *Solanaceæ*, to which the potato and tomato belong, the commonest species being *C. annum* and *C. fastigiatum*, known also under many synonyms.

The first, Flückiger and Hanbury state, furnishes the larger kinds of pod pepper, and, as they believe, much of the cayenne pepper which is imported into England in a state of powder.

C. fastigiatum is the species which is officinal in both the British and United States pharmacopœias. It is grown in tropical Africa and America and appears in our market as Zanzibar pepper. The color of its powder is lighter yellow than that of the preceding. The two species furnish the market with its cayenne pepper, although a few other species are sought in small amount for their peculiar flavor.

Of the microscopic structure Flückiger and Hanbury say :

The pericarp consists of two layers, the outer being composed of yellow thick-walled cells. The inner layer is twice as broad and exhibits a soft shrunken parenchyme, traversed by thin fibro-vascular bundles. The cells of the outer layer especially are the seat of the fine granular coloring matter. If it is removed by an alcoholic solution of potash, a cell nucleus and drops of fat or oil make their appearance. The structural details of this fruit afford interesting subjects for microscopical investigation.



FIG. 12. Cayenne; A, outer epidermis. B, stone cells of the inner epidermis; C, epidermis cells of the edge of seed. (After Schimper.)

The peculiarities described above are so distinctive that the presence of foreign matter is easily detected. The cells of the pericarp or epidermis are of a peculiar flattened and chain-like angular form which are

characteristic of cayenne. The other structures are not as prominent, but are not liable to be confounded with those of any adulterants. Diagrammatic representatives of this structure are given in Fig. 12, and the appearance of the pure ground cayenne under polarized light in Fig. 48, Plate XXIV. The portions of the seed in the powder are not readily distinguished without careful examination. They are, however, characteristic and contain starch, the form of which is shown in Fig. 67, Plate XXVII. The adulterants, which are said by Hassall to be more numerous and frequent than with ordinary pepper, are given by authorities as mineral coloring matter added to hide the loss of color which takes place on exposure of cayenne to light, to add to the weight, or to cover the addition of colorless diluents, ground rice, turmeric, husks of mustard, etc.

Mineral adulterants were met with in but one sample of a low grade, obtained in the Baltimore market. The organic ones, on the contrary, were almost always present, among them yellow corn meal being found, but rice or corn flour oftener. The latter was readily detected with the polariscope, as illustrated in Fig. 49, Plate XXIV. The few starch grains in the lower layers of the pericarp and in the seed are very small and cannot be confused with the added rice or corn. Turmeric and mustard are recognized by their peculiar structure.

Of the chemistry of *Capsicum* but little had been written until 1884, when Strohmer undertook an investigation of the proximate composition of the pod. Bracconot had announced the discovery of the active principle, and named it capscine. Wilting had asserted that it was a crystalline alkaloid. Thresh had discovered a crystalline body capscine, which he regarded as the substance to which the pungency was due, but Strohmer showed that there was present in the cayenne—

(1) A fixed oil, without sharp smell or taste, that required 201.9 of KOH for saponification and occurred almost entirely in the seed.

(2) A camphor-like substance which tastes and smells sharp, and which constitutes the peculiar principle of the cayenne (capsicine). It is contained in the pods and seeds, although in greater amount in the former than in the latter, where it is dissolved in a fixed oil.

(3) A resinous body, the red coloring matter, (capsicum red) which is only contained in the pod.

Quantitative determinations were made of the fruit of *Capsicum annum*, grown in Hungary.

	Seed.	Pod.	Whole fruit.
Water at 100°.....	8.12	14.75	11.94
Albuminoids.....	18.31	10.69	13.88
Fat. (ether extract).....	28.54	5.48	15.26
Nitrogen free extract by difference.....	24.33	38.73	32.63
Crude fiber.....	17.50	23.73	21.09
Ash.....	3.20	6.62	5.20
Total.....	100.00	100.00	100.00
Nitrogen.....	2.93	1.71	2.22

The percentage of water given is probably too high, owing to the volatilization of the camphor-like substance.

The ether extract of the seed was nearly pure fat, that of the pod capsicum red.

As to whether a pepper is adulterated or not, Strohmer considers that these determinations will furnish the evidence. He found in commercial Cayennes :

	(I) Rosen- paprika, prima.	(II) Rosen- paprika, sekunda.	(III) Kö- nig's, paprika.
Water	17.35	14.39	12.69
Albuminoids	14.56	14.31	13.19
Ether extract	14.43	15.06	13.35
Ash	5.10	5.66	7.14

The latter specimen was adulterated with stems, &c., and the results show the agreement between the degeneration in quality and the analytical determinations.

In our investigations several specimens have been examined with the following results :

- 4517. English brand.
- 4545. New York brand.
- 4554. Bulk, corner grocery.
- 4554.¹ Public lunch-room.
- 4880. Baltimore spice mills.
- 4881. Baltimore spice mills.
- 4897. Zanzibar ; pure, whole, from importers.

Microscopic examination.

- 4517. Pure, Crosse and Blackwells. Bottled.
- 4545. Adulterated rice flour.
- 4554. Adulterated rice flour.
- 4554.¹ Adulterated rice flour.
- 4880. Adulterated }
- 4881. Adulterated } yellow corn, turmeric, and red ocher.

The analytical results were :

Serial No.	Water.	Ash.	Fixed oil.	Volatile cam- phor, &c.	Fiber.	Albuminoids.	Undeter- mined.	Total.	Nitrogen.	Quality.
4897.....	2.35	9.06	0.12	26.99	16.88	13.13	41.47	100.00	2.10	Pure.
4517.....	5.74	5.24	1.58	17.90	18.10	11.20	40.24	100.00	1.79	Do.
4545.....	3.70	6.16	3.40	14.50	15.40	10.15	46.75	100.00	1.62	Adulterated.
4554.....	5.20	6.65	1.65	14.11	10.53	9.98	51.88	100.00	1.60	Do.
4880.....	1.41	4.68	4.00	9.41	14.70	7.70	58.10	100.00	1.23	Do.
4881.....	1.93	8.69	3.48	6.66	13.33	7.00	58.91	100.00	1.12	Do.

From the data it is not difficult to detect the presence of adulterants. The whole Zanzibar pepper ground in the laboratory has 26.99 per cent. of non-volatile ether extract, much more than was found by Strohmer, and the other pure specimen 17.90 per cent. None of the others reach these figures. In albuminoids the addition of starchy and mineral diluents also makes a marked difference, their presence being revealed in a striking way.

The unexplained presence of so much volatile matter on the two worst specimens and of so much ash in the Zanzibar of good grade as compared with these same specimens containing mineral adulterants, is remarkable and shows that too hasty conclusions must not be drawn from the chemical data. It should be noticed also that the water in our specimens falls far below that given by Strohmer, a peculiarity which has been noted in other cases. In the detection of adulteration of Cayenne by chemical methods, determinations of water and ash, ether extract and albuminoids are, it seems, of value and as a rule will serve to reveal the means of adulteration. When combined with a microscopic examination the estimation of the amount of the diluents present would not be difficult.

GINGER.

The rhizome, or commonly root, of *Zingiber officinale* is known as ginger. It is a "reed-like plant, with annual leafy stems 3 to 4 feet high, and flowers in cone-shaped spikes borne on other stems thrown up from the rhizome. It is a native of Asia, in the warmer countries of which it is universally cultivated, but is not known in the wild state. It has been introduced into most tropical countries, including the West Indies."

Ginger occurs in two forms—dried with the epidermis as coated ginger and as scraped ginger when the epidermis is removed. The uncoated ginger is prepared by scraping and washing the rhizome and then drying it in the sun. Thus prepared, Flückiger and Hanbury say:

It has a pale buff hue and breaks easily, exhibiting a short and farinaceous fracture with numerous bristle-like fibers. When cut with a knife the younger and terminal portion of the rhizome appears pale yellow, soft, and amylaceous, while the older part is flinty, hard, and resinous.

Coated ginger or that which has been dried without the removal of the epidermis is covered with a wrinkled, striated brown integument, which imparts to it a somewhat coarse and crude appearance, which is usually remarkably less developed on the flat parts of the rhizome. Internally it is usually of a less bright and delicate hue than ginger from which the cortical part has been removed. Much of it, indeed, is dark, horny, and resinous.

In our markets we find Jamaica ginger, which is the finest variety, being very carefully prepared and scraped, and several qualities of brown ginger which is unscraped and not carefully prepared, and is imported from India and the East. The latter is sometimes bleached

and coated with gypsum or carbonate of lime to improve its appearance. This method of treatment appears to be very common.

Of the microscopic appearance of the rhizome Flückiger and Hanbury write as follows :

A transverse section of coated ginger exhibits a brown, horny external layer, about one millimetre broad, separated by a fine line from the whitish mealy interior portion, through the tissue of which numerous vascular bundles and resin cells are irregularly scattered. The external tissue consists of a loose outer layer, and an inner composed of tabular cells; these are followed by peculiar short prosenchymatous cells the walls of which are sinuous on transverse section and partially thickened, imparting a horny appearance. This delicate felted tissue forms the striated surface of scraped ginger and is the principal seat of the resin and volatile oil, which here fill large spaces. The large-celled parenchyme which succeeds is loaded with starch and likewise contains numerous masses of resin and drops of oil. The starch granules are irregularly spherical, attaining at the utmost 40^{mm}. Certain varieties of ginger, owing to the starch having been rendered gelatinous by scalding, are throughout horny and translucent. The circle of vascular bundles which separates the outer layers and the central portion is narrow and has the structure of the corresponding circle or nucleus sheath on turmeric.

In the best ground ginger of the trade little is seen of this structure. The appearance is one of predominating starch grains which are so abundant in the parenchyma, and which are figured in Plate XXI, Fig. 41, and Plate XXVII, Fig. 58. They are so characteristic in shape and in their appearance with polarized light, that they are readily distinguished from adulterations.

Among them are seen scattered the fibro-vascular bundles, the turmeric-like resin bodies, and less prominently the oil globules, which are few in number.

When the source of the powder is the ordinary unscraped ginger, the outer horny layer is prominent, but not distinct in its character, and at times when the rhizome has been scalded the starch grains are swollen and the whole structure is more difficult to make out. It is not, however, as a rule, difficult to detect foreign matter in the powder of this spice.

ADULTERANTS.

The principal adulterants are said to be mineral matter, sago, tapioca, flour of rice, wheat, and potato, cayenne, mustard hulls, turmeric, and exhausted ginger. The foreign starches, cayenne, and mustard hulls, are easily detected, but the turmeric cells, from their resemblance to the resin globules of the ginger, are more confusing. The too frequent occurrence of such cells is suspicious. For the detection of exhausted ginger recourse must be had to proximate analysis. The recommendation also of Hassall, to wash away some of the starch, or the use of a sieve, will be found of advantage, the remaining coarse particles being then seen in greater numbers and with more ease. We have found in the gingers of the trade rice flour, turmeric, and hulls of foreign seeds. But two of the specimens were derived from coated root.

CHEMICAL COMPOSITION.

Owing to the practice of diluting the ground spice with the residues from extraction for turmeric, &c., the normal proximate composition is of importance.

W. C. Young* has examined seven well-authenticated specimens with results which have been reprinted here.

Sources.	Moisture at 100° C.	Ash.	Ash, insoluble in H ₂ O.	Ash, soluble in H ₂ O.	Aqueous extract, 2 per cent. de- coction.	Mucilage.	Alcohol extract, 5 per cent. de- coction.	Resin.	Cellulose
African 1.....	15.8	3.4	1.34	2.06	24.8	18.0	8.5	2.2	5.1
African 2.....	14.5	4.3	1.58	2.72	52.2	15.7
Jamaica.....	15.0	5.4	1.22	4.18	55.7	32.3	6.5	.25	3.1
Cochin.....	15.2	5.8	3.28	2.52	35.1	21.8	12.5	4.5	9.0
Japan.....	15.2	8.0	5.82	2.18	34.3	19.4	8.3	2.8	4.6
Malabar.....	10.2	3.4	1.6	1.8	30.1	22.4	4.1	1.7	1.7
Bengal.....	20.50	4.75	2.36	2.39	51.4	41.1	4.3	.84	4.9

These specimens were all scraped with the exception of the last two.

The analyses do not seem to have been carried out on accepted principles, and are not of great value except for ash and perhaps fiber, but in complete want of any data they have been copied. The fact that the decorticated specimens contain no more fiber than the others is peculiar. The methods of determining "resin" and "mucilage" and the meaning of the terms also are not given. Little is therefore learned from them.

Tresh† has investigated the ether extract of the rhizome, but his results furnish us with no data of analytical use. Lately E. W. T. Jones‡ has, under the title "The Amount of Starch in Ground Ginger," obtained some data which are of value, but unfortunately are confined to only one specimen of ginger. He determined water and ash as usual, extracted with ether, after moistening with alcohol (Sp. gr 0.90), at 35°-38°, and with water at the same temperature. The residue, after gelatinizing the starch, was submitted to diastatic action. In the filtrate from the unacted-upon substance, which was weighed as fiber, the dextrine and maltose were determined by the polariscope. Fiber was also determined by the usual method of treatment with acid and alkali. The results were:

*Analyst, **9**, 214-215, 1884.

† Pharm. J., Trans., **37**, 610, 721, 1882.

‡Analyst, **11**, 75-77, 1886.

	Per cent.
Moisture, loss at 100° C.....	10. 10
Ether extract	3. 58
Alcohol extract	3. 38
Water extract	3. 66
Starch, calc., from dextrine and maltose ..	52. 92
Fiber, residue from diastatic action	19. 12
Crude fiber by acid and alkali	2. 66
Ash	4. 80
Matter not accounted for in the starch products ..	1. 50
	99. 06

Practically we have learned from all that has been hitherto done in the proximate examination of ginger that the ash may vary from 3.4 to 8.0 per cent. in genuine samples, and fiber from 1.7 to 9.0 per cent. Young and Jones differ altogether too much on other points to be of any value. We are therefore in need of further information to enable us to detect the addition of spent ginger as an adulterant, the differences in determinations of fiber being so wide as to render this determination of no value, since it is possible to even reduce the amount of fiber by such an addition and judicious selection.

With a view to extending our knowledge of the subject we have examined a number of pure gingers, obtained from the importers and several samples of ginger found in the local markets, some of which, as was learned from the microscopic examination, were adulterated with farinaceous matter and foreign hulls. The results were :

Analyses of gingers.

Sources of samples :

- 4506. Scraped Jamaica, limed, whole.
- 4889. Whole Calcutta ginger root, unscraped and unbleached.
- 4890. Whole Cochin ginger root, unscraped and unbleached.
- 4891. Whole Jamaica ginger root, unscraped and unbleached.
- 4892. Whole Jamaica ginger root, London market, bleached.
- 4893. Whole Jamaica ginger root, American market, bleached.
- 4507. White ground Jamaica, "pure."
- 4521. Brown, ground in Washington.
- 4526. Brown, ground in Baltimore.
- 4541. Genuine Borneo, English brand.
- 4549. Brown.
- 4242. Mohawk, N. Y.
- 4875. Ground ginger, Baltimore Spice Mills, "pure."
- 4876. Ground ginger, Baltimore Spice Mills, "best."

Microscopical examination:

- 4506 and 4889-4893. Pure standards.
- 4507. Not as much fiber matter as in the preceding, but no adulterants detected.
- 4521. Good brown ginger.
- 4526. Starch, turmeric, and foreign husks.
- 4541. Good and white.
- 4549. Starch, and perhaps turmeric ; brown.
- 4242. Starch and some questionable substance.
- 4875 and 4876. Cereals and cayenne; perhaps exhausted ginger.

Analyses of gingers.

[Whole ginger root]

Serial No.	Source.	Water.	Ash.	Volatile oil.	Fixed oil and resin.	Starch.	Crude fiber.	Albuminoids.	Undetermined.	Total.	Nitrogen.
4889	Calcutta	9.60	7.02	2.27	4.58	49.34	7.45	6.30	13.44	100	1.01
4890	Cochin	9.41	3.39	1.84	4.07	53.33	2.05	7.00	18.91	100	1.12
4891	Unbleached Jamaica	10.49	3.44	2.03	2.29	50.58	4.74	10.85	15.58	100	1.74
4892	Bleached Jamaica, London	11.00	4.54	1.89	3.04	49.34	1.70	9.28	19.21	100	1.48
4893	Bleached Jamaica, American	10.11	5.58	2.54	2.69	50.67	7.65	9.10	11.66	100	1.46
4506	Bleached Jamaica	9.10	4.36	.96	3.09	46.16	3.15	5.25	27.93	100	.84

[Commercial ground.]

Serial No.	Source.	Quality.	Water.	Ash.	Volatile oil.	Fixed oil and resin.	Starch.	Crude fiber.	Albuminoids.	Undetermined.	Total.	Nitrogen.
4507	Bleached	Pure	8.06	3.47	1.78	3.11	48.92	2.65	6.13	25.88	100	.98
4521	Unbleached	Pure	11.20	6.02	1.61	4.12	50.00	4.08	6.28	16.69	100	1.04
4526	do	Adulterated	10.35	5.83	1.52	4.66	46.49	3.48	8.75	18.92	100	1.40
4541	do	Pure	8.90	3.45	.95	3.65	49.12	3.45	6.30	24.18	100	1.01
4549	do	Adulterated	9.45	4.75	1.45	4.30	50.58	4.20	8.93	16.34	100	1.43
4875	Brown	Pure	11.82	7.34	2.61	3.21	46.49	4.98	7.35	18.20	100	1.18
4876	do	do	11.33	7.94	2.11	3.51	49.12	3.20	7.35	15.44	100	1.18

From these results it appears that the ash is quite variable, a pure sample having as much as 7.02 per cent. and as little as 3.44 per cent., the whiter varieties having the least and the brown the most. From the ether extract little can be learned. There is no distinction even between the pure and adulterated, the adulterations having a very similar amount of extract. Of the pure samples the white has the least, as well as the least ash. Of fiber the least, as would naturally be expected, is found in the whitest and best scraped and prepared varieties. Of albuminoids it may be said that there is no greater variation in the pure samples than in those which proved to be adulterated. When foreign hulls are present the amount may be much larger. The determinations of starch do not show that the samples have been diluted with cereals or any farinaceous matter, neither could any be detected under the microscope.

The chemical examination proves consequently most unsatisfactory, and it is only possible to give the following hints for pure ginger :

	Per cent.
Water	11.00 to 9.10
Ash	7.02 to 3.39
Volatile oil	2.54 to .96
Fixed oil	4.58 to 2.29
Starch	53.33 to 46.16
Crude fiber	7.65 to 1.70
Albuminoids	10.85 to 5.25

In our markets ginger is drier than abroad, as is usually the fact with all such substances in our climate. The percentage of ash among Young's samples reached 8.0, but did not go below our lowest determination, and our variations in fiber include his figures and those of Tresh.

In other respects our determinations agree with those of the two investigators mentioned, and from their increased number serve as a fair basis for comparison.

A careful qualitative examination of the character of the extracts at times may reveal the presence of an adulterant, but the chief dependence must, it seems, in the examination of this spice, be placed upon the microscope. This, however, will not reveal the presence of exhausted ginger, and a careful study of the effect of exhaustion on the proximate composition of the ground root is therefore desirable. It would naturally increase the relative of percentages of fiber and albuminoids and starch and diminish that of extractive matter.

CINNAMON AND CASSIA

Cinnamon and cassia are the barks of several species of the genus *Cinnamomum*, the true cinnamon (*C. zeylanicum*) being a native of Ceylon, where it is also largely cultivated, while the cassias are derived from several species growing in Bengal and in the countries east of India, especially China and in the Indian Archipelago. In our markets the Chinese and Malabar cassias occupy the most prominent place, Batavia and various other localities supplying a poorer quality. Ceylon cinnamon is only found in small amount and usually is in use as a drug. In our experience it is not to be procured in a ground condition. In their original form there is no difficulty in identifying the various barks by their peculiar characteristics. Ceylon cinnamon, during its preparation, is deprived of its outer corky coat and of the inner cortical layers. It is therefore thin, not more than one-eighth to one-sixteenth inch thick, and is distinguished by having the quills, which curl inward on each side, arranged one within the other, forming a flattened cylinder, originally a yard long. Its color is a dull brown, and the removal of the outer coats is discovered by the appearance on the exterior of the peculiar wavy structure of the inner layers of the bark.

Cassia, on the other hand, commonly shows the outer corky coat of the bark, and is, in consequence, much thicker, rougher, and of not so red color. In some cases, however, the bark is scraped, but the character of the surfaces are still distinctly marked, and it is thicker and deeper in color than cinnamon.

Microscopically, true cinnamon consists of thin layers, which in the ground drug are represented most prominently by the long cells of woody fiber or liber fibers which are scattered through the bark, and which are distinctly seen under polarized light, the stellate cells of the outer layers and the thinner cells of the interior carrying the few starch grains. In cassia, which of course bears a close general resemblance

to cinnamon, the woody fiber is comparatively small in amount, as is the number of the stellate cells. The presence of starch is, on the other hand, more marked. A view, therefore, of a true ground cinnamon and cassia with polarized light reveals at once differences which are characteristic enough to distinguish the specimens, and which are illustrated in figures 46 and 47, plate XXIV. In the ground specimens which we have had in hand, however, no specimens of true cinnamon have been found. Reference was made, therefore, to ground samples prepared from authentic specimens of Ceylon and Chinese barks, and before any microscopic work is attempted it is necessary to make a comparative examination of this description to acquire a knowledge of the appearances to be expected and which cannot be described in detail. Reference may also be usefully made to Flückiger and Hanbury, *Pharmacographia*, for many details in regard to the growth, cultivation, preparation, &c.

Of the addition of cedar sawdust, roasted hulls, oil meals, and mineral and coloring matter we have had but little opportunity to judge. Adulteration in these markets is mainly confined to replacing cinnamon by cassia, and the adulterants mentioned are found only in the lowest grades, while their entire difference in structure make their detection microscopically a matter of no difficulty.

Schimper in his *Anleitung zur mikroskopischen Untersuchung der Nahrungs- und Genussmittel*, describes in detail the appearance of these foreign substances which in Germany seem to be often used and which Wood in Massachusetts has detected in the forms of ground crackers and nut shells, and in Canada are represented by ground peas and wheat, flour, and, as we have found in Baltimore, by minerals and coloring matter. As will be shown, the determination of fiber and ash are valuable means of discovering the presence of such additions.

CHEMICAL COMPOSITION AND REACTION.

Of the proximate composition of any of the barks but little is known. Numerous determinations and analyses of the ash have been made with a view to detecting peculiarities or the addition of mineral matter.

Hilger* has recently found in five samples of Ceylon cinnamon the following amounts of ash :

	Soluble—per ct.
(1)—4.5	53.0
(2)—4.8	72.3
(3)—3.9	88.1
(4)—4.3	61.7
(5)—3.4

Hehner† in 1879 made an extended investigation into the substitution of cassia for cinnamon, and examined the iodine test, finding it useless, as has been the case when employed in our laboratory. He then investigated the mineral constituents, thinking that the more woody cassia

*Arch. d. Pharm., **223**, 826.

†Analyst, **4**, 223-228.

bark would contain more lime and magnesia than the more delicate cinnamon. Some of his figures are as follows :

Kind.	Price per pound, retail.	Moisture.	Ash in bark.	Lime in ash.	Mn ² O ₃ .	Soluble ash.	Insoluble ash.
	s. d.						
Whole cinnamon	1 10	12.67	4.78	40.09	.86	25.04	74.96
	3 0	12.05	4.59	36.98	.97	28.98	71.02
	3 6	11.38	4.66	40.39	.13	25.22	74.78
	3 6	11.64	3.44	34.32	.62	26.36	73.64
	8 6	12.94	4.28	36.99	.59	27.67	72.33
Cinnamon chips.....	*9	11.25	4.44	42.11	.34	18.34	81.66
Cassia lignea:							
Whole		14.22	1.84	25.29	5.11	40.58	59.42
		11.88	2.54	34.49	4.94	26.78	72.22
Ground		11.05	2.55	28.63	3.55	30.91	69.09
		10.37	4.08	52.72	1.13	8.36	91.64
Cassia vera		11.36	4.85	43.40	1.53	15.89	84.11

*Containing wood.

From these analyses Hehner concludes that the ash in cinnamon varies between comparatively narrow limits. *Cassia vera*, or Malabar cassia, contains as much as cinnamon, *Cassia lignea* less; that one quarter of the ash is soluble in water in cinnamon, less in *C. vera*, and still less in *C. lignea*; that cinnamon ash contains less than 1 per cent. of oxide of manganese, *C. vera* more than 1 per cent., and *C. lignea* far more, even up to 5 per cent. This is the most noteworthy feature and is shown in the color of the ash, cinnamon ash being white, while those of cassias are gray or brown. *Cassia vera*, the Malabar cassia, is not so readily distinguished from cinnamon as *C. lignea*, but its occurrence is less frequent. The percentages of lime and magnesia were not distinctive, as had been expected. For cinnamon, 40.09, 36.98, and 40.39 were the amounts of lime in three specimens. In one of *Cassia lignea* 25.29 per cent. was found. In one of *C. vera*, 52.72. The less fibrous *C. lignea* contained the least lime. Of magnesia there was 2.65, 3.30, and 3.86 in the cinnamon and 5.48 and 1.10 in the *Cassia lignea* and *vera* respectively. Little seems to have been learned, therefore, which would form a sound basis for distinguishing these barks. The presence of manganese is more or less accidental and cannot be considered as an essential element of the ash or one from which such definite conclusions could be drawn as to serve as the basis of legal testimony. Our results, in fact, failed to show any large amount in any of the specimens.

Of the proximate organic constituents, the presence of volatile oil, tannin, mucilage, coloring matter, resin, an acid, starch, and lignin is noted by Hassall, without any details in regard to their proportions. The essential oil, according to Pharmacographia, however, amounts to only one-half to 1 per cent. of the bark of cinnamon and must be less in the inferior cassias. Beyond this no data are found. We have examined a number of specimens of the ground bark bearing different

designations and three or four samples of bark obtained unground and several authenticated specimens direct from the importers. The sources and descriptions of the material examined were:

DENOMINATED CINNAMON.

- 4502. Bark from grocers, D. C. Poor quality.
- 4503. Ground, from same grocers. Both guaranteed, pure.
- 4519. Ground in Washington, 30 cents per $\frac{1}{2}$ pound.
- 4529. Ground in Baltimore, 20 cents per $\frac{1}{2}$ pound.
- 4531. Ground in Baltimore, 30 cents per $\frac{1}{2}$ pound.
- 4539. Genuine Java cinnamon (cassia), English, 10 cents per $\frac{1}{2}$ pound.
- 4547. Cinnamon, Java, ground in Washington.
- 4556. Ceylon cinnamon bark. Druggists.
- 4558. Ceylon cinnamon bark. Druggists, 15 cents per ounce.
- 4868. Cinnamon, pure, Baltimore, Md.
- 4869. Cinnamon, best, Baltimore, Md.

DENOMINATED CASSIA.

- 4557. Cassia bark. Druggists.
- 4559. Cassia bark, 10 cents per ounce. Druggists.
- 4640. Cassia bark, 10 cents per ounce. Druggists.
- 4906. Saigon cassia chips, collected in Baltimore markets by Z. D. Gilman.
- 4907. Cassia ligna bark, collected in Baltimore markets by Z. D. Gilman.
- 4908. Batavia bark, collected in Baltimore markets by Z. D. Gilman.
- 4909. Saigon cassia bark, collected in Baltimore markets by Z. D. Gilman.

A mechanical and microscopic examination showed that the ground specimens were hardly what they were represented to be.

4503, guaranteed pure cinnamon, proved to be a good quality of ground cassia.

4519 and 4529 consisted of substitution of cassia for cinnamon.

4531 was a low grade cassia mixed with considerable foreign material.

4539 was an excellent specimen of good cassia.

4547 was a poor quality cassia, adulterated with foreign material, but so finely ground as to make its identification impossible.

4868 and 4869, although labeled cinnamon, were mixtures of the lowest grade, consisting of cassia, turmeric ochre in small amount, mustard hulls or those of a similar seed, cracker dust, and burnt shells.

The remaining specimens were purchased unground, and, with the exception of the one English brand labeled Java cinnamon (cassia), no ground cassia could be purchased under such a designation.

The data obtained in a chemical way were as follows:

Analyses of cinnamon and cassia.

Serial No.	Description.	Sold as—	Consisting of—	Price per pound.	Water.	Ash.	Volatile oil.	Fixed oil, &c.	Crude fiber.	Albuminoids.	Undeter- mined.	Total.	Nitrogen.
4502	Ceylon bark.....	Cents.	10.00	3.70	3.14	3.30	16.18	3.80	59.88	100	.61
4556	do.....	*15	5.40	4.55	1.05	1.66	33.08	2.98	51.28	100	.48
4558	do.....	*15	7.93	3.40	.82	1.58	25.63	3.80	56.84	100	.62
4557	Cassia bark.....	9.42	2.35	.58	1.40	17.73	2.80	63.72	100	.45
4559	do.....	9.01	1.75	.84	1.75	20.63	2.45	63.57	100	.39
4640	Buds.....	4.79	3.58	3.59	5.21	8.60	7.00	65.23	100	1.12
4906†	Cassia bark.....	10	8.23	1.01	2.13	26.29	4.20	48.65	100	.67
4907	do.....	11.04	2.48	1.21	1.86	15.45	2.63	65.33	100	.42
4908	do.....	17.45	5.25	.55	.74	14.33	4.03	63.65	100	.64
4909	do.....	9.32	5.86	3.51	2.38	16.95	4.55	57.43	100	.73
4503	Commercial ground bark..	Cinnamon	Cassia	5.00	4.80	3.70	3.92	29.88	2.00	56.10	100	.33
4519	do.....	do	do	60	9.37	3.36	.96	2.46	18.85	2.28	62.72	100	.36
4529	do.....	do	do	40	9.51	3.42	1.19	2.69	15.73	2.63	64.83	100	.42
4531	do.....	do	do	40	7.76	2.92	1.37	1.92	16.43	2.98	66.62	100	.48
4539	do.....	do	Cassia	1.20	5.19	5.65	4.41	3.70	19.10	3.15	58.80	100	.50
4547	do.....	Cassia	C. mixtures.	40	6.45	2.50	1.00	1.96	18.43	2.10	63.56	100	.34
4868	do.....	do	do	16	8.73	4.02	1.90	2.86	16.88	4.55	61.06	100	.73
4869	do.....	do	do	14	11.21	3.74	1.15	2.13	19.18	4.20	58.39	100	.67

* Per ounce.

† Saigon chips.

‡ Lignea bark.

§ Batavia bark.

|| Saigon bark.

The preceding figures show that there is nothing particularly distinctive between the cinnamons and cassias, and that the determination of volatile oil points more to the character and value of the bark than any other, though, at the same time, there is nothing distinctive therein, as the variation in amount is so large in the cinnamons as to at times furnish samples containing far less volatile oil than even a fair cassia. The percentage of ash is extremely variable, depending on the age and quality of the bark. Saigon chips were found to have 8.23 per cent., while a specimen of unidentified cassia bark had only 1.75 per cent. Cinnamon bark will probably average less than cassia. Fiber, like ash, is extremely variable, and for the same reasons; 26.29 per cent. were found in Saigon chips and 33.08 per cent. in a cinnamon and from 14 to 20 in ordinary cassias. This determination, therefore, reveals nothing, and is of no assistance in detecting adulterants.

Albuminoids are variable, but within narrow limits, the extremes being 4.55 percent. in Saigon bark and 2.45 in an unidentified cassia. The Batavia and Saigon barks appear to contain the most, over four per cent., and this percentage would seem to be an indication of *inferior quality*.

The amount of tannin in these barks is extremely small, not reaching in our specimens an equivalent of quercitannic acid by the Löwenthal process of two per cent. The addition, therefore, of material containing tannin can be readily detected, but in no case under our observation did such an addition occur.

Aside from the determination of volatile oil, chemical analysis seems to be of little value. The principal dependence must, with our present knowledge, be placed on the mechanical and microscopic examination, since the worst mixtures, 4868 and 4869, scarcely revealed in their composition the fact of their inferiority.

CLOVES.

Cloves are the flower-buds of an evergreen tree, *Eugenia caryophyllata*, growing wild in the Malaccas and introduced into Amboyna, the neighboring Zanzibar islands, Cayenne, and a few other places in the tropics. They are picked by hand when their development has reached a red color, and are dried in the sun, becoming dark brown. They are classed as East Indian, African, and American, and are valued in that order.

Flückiger and Hanbury's description of them is as follows:

Cloves are about six-tenths of an inch in length, and consist of a long cylindrical calyx dividing above into four pointed spreading sepals which surround four petals, closely imbricated as a globular bud about two-tenths of an inch in diameter.

The petals, which are of a lighter color than the rest of the drug and somewhat translucent from numerous oil cells, spring from the base of a four-sided epigynous disc, the angles of which are directed toward the lobes of the calyx. The stamens, which are very numerous, are inserted at the base of the petals and are arched over the style. The latter, which is short and subulate, rises from a depression in the center of the disc. Immediately below it, and united with the upper portion of the calyx, is the

ovary, which is two-celled and contains many ovules. The lower end of the calyx (hypanthium) has a compressed form; it is solid, but has its internal tissue far more porous than the walls. The whole calyx is of a deep rich brown, has a dull wrinkled surface, a dense fleshy texture, and abounds in essential oil, which exudes on simple pressure with the nail.

The varieties of cloves occurring in commerce do not exhibit any structural differences. Inferior kinds are distinguished by being less plump, less bright in tint, and less rich in essential oil. In London price-currents cloves are enumerated in the order of value thus: Penang, Bencoolen (Sumatra), Amboyna, Zanzibar.

A transverse section of the lower part of a clove shows a dark rhomboid zone, the tissue on either side of which is of a lighter hue. The outer layer beneath the epidermis exhibits a large number of oil cells, frequently as much as 300 micromillimeters in diameter. About 200 oil cells may be counted in one transverse section, so that the large amount of essential oil in the drug is well shown by its microscopic characters. The above-mentioned zone is chiefly made up of about 30 fibro-vascular bundles, another stronger bundle traversing the center of the clove. The fibro-vascular bundles, as well as the tissue bordering the oil cells, assume a greenish-black hue by alcoholic perchloride of iron. Oil cells are also largely distributed in the leaves, petals, and even stamens of *Eugenia*. No starch is found in it, however.

Preparations from whole cloves enable one to familiarize himself with the structure, which in the ground cloves is recognized with greater difficulty. In both cases the use of chloral hydrate solution is desirable, as the sections and fragments are otherwise not transparent. Preliminary examination of the powder in water for starch should, however, be made, as the starch granules swell in the chloral hydrate solution.

Among the fragments will be seen a large amount of débris of no apparent structure, but the larger pieces are chiefly the cells of the epidermis interspersed with the large oil cavities or cells, which are not as readily detected as in carefully prepared sections, being concealed by a layer of the epidermal cells. Next in prominence are the fibro-vascular bundles with their spiral vessels and with shreds of deep brown cellular matter attached. Pollen grains and, at times, whole anthers are present, and concretions of oxalate of lime. All these characteristic appearances are made out much more easily under polarized light, the long cells of the fibro-vascular bundles being optically active, as are also the pollen grains, oxalate of lime to a less degree, and the contents of the oil cells which are thus easily distinguished.

After a study of standard powder of cloves, the presence of adulterants is not difficult to recognize. Pimento is often added, and may be identified by the starch which it contains and the characters described under that spice.

Clove stems are said to be the commonest adulterant, and the presence in them of thick-walled stone cells and long yellow fibrous tissue serves as a means of recognition, since similar structures are not found in the clove, at least in the same abundance. The fruit of the clove is also added, and since it contains starch and a large embryo they are detected readily. We have not met either of these adulterants.

In the cheaper forms of ground cloves, where from the price it is evident that adulteration has been practiced, the common substitutes, which are added as diluents to all the spices, must be sought for and require no further experience beyond what has been obtained in the examination of pepper.

In most of the samples which were examined during the preparation of this report the microscope revealed nothing foreign, and for a decision as to their quality it was necessary to have recourse to chemical analysis. Two specimens from Baltimore of the same low grade as several of the spices previously mentioned were found, however, to be mixtures of all sorts of cheap material, containing mineral coloring matter, roasted shells or charcoal, corn, and hulls of seeds.

The sources of the cloves examined were as follows, being largely the ordinary article for sale at grocers :

SOURCES OF CLOVES.

- 4504. Whole cloves, guaranteed, Washington.
- 4903. Amboyna cloves, whole, direct from importers.
- 4904. Singapore cloves, whole, direct from importers.
- 4905. Clove stems, whole, direct from importers.
- 4641. Whole cloves, druggists', 10 cents per ounce.
- 4642. Whole cloves, druggists', 10 cents per ounce.
- 4643. Whole cloves, druggists', 10 cents per ounce.
- 4505. Ground cloves, guaranteed, Washington.
- 4520. Ground gloves, ground in Washington.
- 4540. Ground cloves, genuine Amboyna, 30 cents per $\frac{1}{2}$ pound ; English brand.
- 4548. Ground cloves, Washington grocers, second class, 26 cents per $\frac{1}{2}$ pound.
- 4629. Ground cloves, Washington grocers, second class, 20 cents per $\frac{1}{2}$ pound.
- 4630. Ground cloves, Washington grocers, 30 cents per pound.
- 4631. Ground cloves, "strictly pure," 40 cents per pound.
- 4632. Ground cloves, Washington grocers, second class, 30 cents per pound.
- 4633. Ground cloves, Washington grocers, second class, 30 cents per pound.
- 4873. Ground cloves, Baltimore Spice Mills ; "pure."
- 4874. Ground cloves, Baltimore Spice Mills ; "best."

The results of the analyses are as follows :

Analyses of cloves.

[Whole cloves.]

Serial No.	Source.	Weight of 100 cloves in grams.	Quality.	Water.	Ash.	Volatile oil.	Fixed oil and resin.	Crude fiber.	Albuminoids.	Nitrogen.	Oxygen equivalent.	As quercitanic acid.	Price per $\frac{1}{4}$ pound.
4504	Grocers.....	6.95	5.99	16.35	7.12	9.75	4.73	.76	4.06	15.86
4641	Druggists.....	3.98	9.31	16.61	9.72	6.94	6.48	1.04	4.73	18.46	\$0 40
4642do.....	5.96	7.66	10.23	9.94	8.70	6.48	1.04	5.13	20.02	40
4643do.....	2.90	13.05	15.87	10.07	8.55	7.00	1.12	3.00	11.70	40
4751	Extra quality.....	10.505	8.67	7.72	17.94	9.54	7.83	5.60	.90	5.43	21.19
4903	Amboyna.....	8.78	5.25	18.89	10.24	6.18	5.42	.87	4.80	18.72
4904	Singapore.....	8.710	10.67	5.50	13.52	9.95	9.08	5.42	.87	5.67	22.13
4905	Stems.....	10.18	6.96	4.40	4.03	13.58	5.78	.92	5.96	23.24

[Commercial ground cloves.]

4505	Fair	5.93	5.79	13.93	6.74	9.38	5.34	.86	5.60	21.84
4520	Poor quality.....	8.86	5.98	9.88	6.58	11.75	4.20	.78	4.80	18.72	15
4540do.....	8.30	5.85	12.40	6.58	11.10	4.38	.70	5.40	24.18	15
4548do.....	8.79	6.05	10.21	7.44	10.93	4.38	.70	5.40	21.84	13
4629do.....	8.78	8.48	8.45	5.36	10.75	5.95	.95	6.07	23.66	10
4630do.....	9.58	10.73	6.52	4.94	12.30	5.78	.92	4.90	19.11	*30
4631do.....	7.48	7.75	12.67	6.74	10.00	6.48	1.04	5.20	20.58	*40
4632do.....	8.71	7.75	3.94	4.02	13.80	5.95	.95	2.89	11.28	15
4633do.....	7.99	8.75	7.31	5.20	12.25	5.60	.90	5.33	21.58	10
4873	Adulterated.....	11.00	9.59	4.06	4.46	17.58	5.42	.87	3.00	11.70	*24
4874do.....	8.62	8.35	3.59	4.47	17.78	5.08	.81	2.96	11.54	*21

* Per pound.

In comparison with these results it is of interest to refer to what has been already published.

Fluckiger states that from 16 to 20 per cent. of volatile oil is present, and gives details in regard to its composition and reactions; but beyond this nothing.

Dietsch gives the following figures as the percentages of oil to be expected in cloves from different sources:

	Per cent.
Amboyna.....	16 to 21
Zanzibar	12 to 17
Cayenne.....	9 to 12

There are one or two very old analyses in detail which are hardly reliable, and Dr. Ellis, of Toronto, has made investigations, still unpublished, in regard to the amount of tannin present, with a view to using the determination as a check on adulterants; but with these exceptions we are not aware of any other work upon the composition of this spice.

The authentic whole samples show that the percentage of water may be very variable, being at times as low as 2.90 per cent., and again as much as 10.67, which is high for so oily a substance. The ash, too, has rather wide extremes, varying from 5 to 13 per cent. The usual amount would not, however, be far from 5.50 to 6.50 per cent. Volatile oil falls in no case below the amount given by Dietsch, and serves as, perhaps, the best means of judging of the quality of the specimens. The extremes found in the pure specimens were 10.23 and 18.89 per cent., while but five out of the eleven ground specimens reached 10 per cent.

Other determinations do not seem especially characteristic. The extremes, which cannot be exceeded without casting suspicion, are:

	Per cent.
Water	11. 00 to 2. 75
Ash	13. 00 to 5. 00
Volatile oil	21. 00 to 9. 00
Fixed oil and resin ...	11. 00 to 4. 00
Crude fiber.....	10. 00 to 6. 00
Albuminoids.....	8. 00 to 4. 00

The determination of tannin, following the suggestions of Dr. Ellis, of Toronto, has been examined with results showing it to be of some value, but not as great as that of volatile oil. Our experience showed that it was as well to determine the matter oxidizable by permanganate, after removal of oil, &c., by Squibb's ether, as to make a more elaborate determination of tannin itself. In the best whole cloves from the importers the quercitannic acid equivalent of the oxidizable matter varied from 18.72 to 22.13 per cent., and in the stem reached 23.24 per cent. The determination will not, therefore, show the presence of stems in ground cloves. The amount fell also in one unidentified specimen of whole cloves to 11.70 per cent., but the quality of these buds was unknown. It is fair to assume, then, that good cloves should contain ex-

tractive oxidizable matter equivalent to 18 per cent. of quercitannic acid, or require an oxygen equivalent of about 4.50 per cent. for its reduction.

Of the 11 specimens of ground cloves examined, although none of them were of first quality, this tannin equivalent exceeded 18 in all but 3. The addition of stems and allspice would not be discovered, as both contain tannin in considerable amount. This determination is, then, in no way conclusive, but merely furnishes an indication which must be corroborated by other means. For the method of carrying it out reference must be made to our pages on analyses. As has been said, none of the ground specimens of cloves were first class, analyses showing that in only one case did the essential oil reach as high as 13.93 per cent., and although but 2 from Baltimore contained cheap foreign adulterants and none were sophisticated with allspice, all the specimens must have been made from a low grade of buds and many with the addition of large quantities of stems and spent cloves. The two cheapest specimens, 4873 and 4874, were, as has been said, terrible compounds of mineral coloring matter, leaving a dark ferruginous ash, corn meal, and hulls, evidence of which appears in the analyses from the low oil, 3.59 and 4.06 per cent., and the high crude fiber. The addition of so much organic matter low in ash conceals the presence of mineral coloring matter which is detected by its ferruginous appearance. In the ash of the whole buds, while there are at times some of a light reddish tinge, the color is distinctly or often dark green from the presence of manganese.

Our results show the universal and alarmingly poor quality of the commercial supply of ground cloves.

PIMENTO OR ALLSPICE.

Pimento is the fruit of *Pimenta officinalis*, an evergreen tree common in the West Indies. It is the only one of the common spices which had its origin in the New World. It is a small, dry, globular berry from two to three-tenths of an inch in diameter, having a short style and surrounded by four short thick sepals which often, however, have become rubbed off, leaving a scar-like ring. The berry has a woody shell, or pericarp, easily cut, and of dark, ferruginous brown, rugose by means of minute tubercles filled with essential oil. It is two-celled, each cell containing a single seed. The seed is less aromatic than the pericarp.

Under the microscope the outer layer of the pericarp, just beneath the epidermis, appears as a collection of very large brown parenchymatous cells filled with oil. The more interior layers consist of thick walled or stone cells loaded with resin, the most characteristic structure of the pimento, parenchyma cells, and smaller crystals of calcic oxalate which are not easily seen. The whole tissue is traversed, but not plentifully, by fibro-vascular bundles. The seeds contain much starch in minute grains and have a few oil cells. The embryo is large and spirally curved. The hulls of the seeds consist of a delicate epidermis

and of large thin-walled cells with light or dark red contents, which are very characteristic and are called by Hassall the port-wine cells, which should be examined in water, and after treatment with chloral hydrate, the starch grains being made out in the water preparation and the remaining structure among the particles rendered transparent by the chloral hydrate. Most prominent among the latter under polarized light, which is here a great assistance, are the stone cells or thick walled cells partly grouped and partly separate, and often, with plain light, showing shreds of parenchyma adherent to them. The brown cells which contain the oil are made out with less distinctness, but most striking are the red or port-wine cells of the seed hull, which are seen scattered everywhere, and in color and form are very characteristic. Shreds of the embryo are also now and then seen.

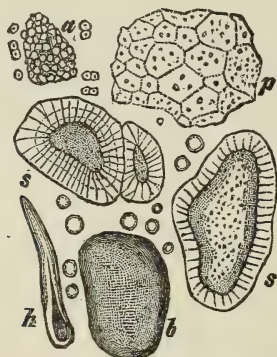


FIG. 13. Allspice powder. *a*, starch; *b*, port-wine cells; *h*, hairs; *s*, stone cells; *p*, parenchyma. $\times 240$.

Schimper's diagrammatic illustrations of this spice are here copied in Fig. 13, and serve as a slight aid to the recognition of the structures mentioned, but merely as suggestions, as nothing exactly corresponding to the drawings will be found in the ground powder.

Polarized light is a most important aid in examining this spice. It brings out strongly the stone-cells and ligneous tissue and differentiates therefrom the great mass of other matter. It also makes the oil cavities more distinct.

The adulteration of this spice does not often occur, owing to its cheapness. We have only found three cases, and those from Baltimore. In No. 4530 a substitution had been made for allspice, of which not a particle could be detected, of inferior cloves from which much of the volatile oil had been extracted, and Nos. 4877 and 4877, where yellow corn and mineral coloring matter were plentiful. In these instances chemical analysis confirmed the microscopic examination.

Abroad clove stems are said to be largely used as an adulterant. They differ from cloves, as has been already explained, in the presence of

numerous stone-cells and fibro-vascular bundles, and in Canada peas and roasted corn have been found. The presence of these cheap diluents should be sought for, as in peppers, but they are less common in this spice.

CHEMICAL COMPOSITION OF ALLSPICE.

But little has been published in regard to the proximate principles of the pimento. The amount of volatile oil is said by Flückiger to be from 3 to 4½ per cent., while starch and much tannin are present in the berry. Dragendorff has also found a minute quantity of an alkaloid of the odor of coumarine. Hassall gives analyses, which are too old to be of any value, and quotes Pereira as an authority for the fact that the essential oil of pimento is made up of two distinct oils, which, of course, may be true in one sense but is hardly of value as a definite distinction, both for this spice and cloves, as has been already remarked.

As a matter of fact but little is known of this spice. We have examined seven samples with the results here given :

4500. Whole allspice.

4501. Ground allspice, guaranteed pure, Washington.

4518. Ground allspice, ground in Washington.

4525. Ground allspice, ground in Baltimore.

4530. Ground allspice, ground in Baltimore.

4534. Ground allspice, ground in Baltimore.

4538. Ground allspice, English brand.

4877. Ground allspice, best, cheap grade, Baltimore.

4878. Ground allspice, pure, cheap grade, Baltimore.

All the samples were ground by different firms except the last two.

Analyses of pimento, allspice.

Serial No.	Description.	Water.	Ash.	Volatile oil.	Fixed oil.	Undetermined.	Crude fiber.	Albuminoids.	Nitrogen.	Tannin equivalent.	O required.
4500	Whole	6.19	4.01	5.15	6.15	59.28	14.83	4.38	.70	10.97	2.81
4501	Commercial ground	5.51	3.93	2.93	6.10	58.24	17.95	5.34	.86	13.10	3.36
4518do	8.03	4.83	2.07	5.50	57.20	18.00	4.38	.70	9.31	2.39
4525do	8.82	4.91	3.32	6.18	57.90	13.45	5.42	.87	9.39	2.40
4530do	11.59	6.02	8.17	7.64	59.57	11.93	5.08	.81	18.72	4.80
4534do	7.31	3.45	3.16	6.92	58.58	16.55	4.03	.64	12.74	3.27
4538do	8.71	4.50	1.29	5.35	55.90	18.83	5.42	.87	10.92	2.60
4877do	7.98	5.53	2.80	3.77	56.86	18.98	4.38	.70	8.27	2.12
4878do	7.31	5.19	1.82	1.60	56.45	23.60	4.03	.67	4.32	1.11

In our analyses there is considerable variation among the samples. Taking the whole berry as the standard the others fall off very much in quality, as judged by the volatile oil, with the exception of number 4530, which, too, from its low fiber, high ash, and high volatile and fixed oil is suspicious, and proves to be a substitution of cloves and clove stems. The microscope has shown that this sample is inferior cloves, and it must therefore be rejected.

The amount of essential oil in our best sample exceeds Flückiger's highest, but on the ground specimens falls off sadly, 4538 and 4878 being almost worthless as far as this valuable ingredient goes, perhaps in the first case having been exhausted for the preparation of the oil, although the ash is but little reduced in amount, and in the latter being nothing but mixtures, as shown under the microscope.

The other determinations show no important variation in the constituents, and estimation of the volatile oil would perhaps be the only thing necessary in a chemical way in examining it, without it is desired to go into the determination of tannin, which is as serviceable a means of discriminating among allspices as was found to be the case with cloves.

In the same way our results show that good allspice contains oxidizable matter extracted by water after removal of oil, etc., by ether equivalent to from 9–11.0 per cent. of quercetannic acid, the amount being considerably smaller than is found in cloves. This determination points out at once that specimens 4530 and 4878 are abnormal, and that 4534 is suspicious. It may be made of value and must be carried out in the same way as with cloves. Of the eight ground specimens examined three were adulterated and one was suspicious, so that even of this cheap spice we can hardly expect a pure supply without some protection.

NUTMEG.

Nutmegs are the interior kernel of the fruit of *Myristica fragrans*, a tree growing in various parts of the East, but principally in the Banda Islands.

Flückiger and Hanbury describe in a most excellent way their characteristics as follows :

The fruit is a pendulous, globose drupe, about 2 inches in diameter, and not unlike a small round pear. It is marked by a furrow which passes around it, and by which at maturity its thick fleshy pericarp splits into two pieces, exhibiting in its interior a single seed, enveloped in a fleshy foliaceous mantle or arillus, of fine crimson hue, which is *mace*. The dark brown shining ovate seed is marked with impressions corresponding to the lobes of the arillus; and on one side, which is of paler hue and slightly flattened, a line indicating the raphe may be observed.

The bony testa does not find its way into European commerce, the so-called nutmeg being merely the kernel or nucleus of the seed. Nutmegs exhibit nearly the form of their outer shell with a corresponding diminution in size. The London dealers esteem them in proportion to their size, the largest, which are about one inch long by eight-tenths of an inch broad, and four of which will weigh an ounce, fetching the highest price. If not dressed with lime, they are of a grayish brown, smooth yet coarsely furrowed and veined longitudinally, marked on the flatter side with a shallow groove. A transverse section shows that the inner seed coat (endopleura) penetrates into the albumen in long narrow brown strips, reaching the center of the seed, thereby imparting the peculiar marbled appearance familiar in a cut nutmeg. * * *

The tissue of the seed can be cut with equal facility in any direction.

Of the microscopic structure they say :

The testa consists mainly of long, thin, radially arranged rigid cells, which are closely interlaced and do not exhibit any distinct cavities. The endopleura which

forms the adhering coat of the kernel and penetrates into it, consists of soft walled, red brown tissue, with small scattered bundles of vessels. In the outer layers the endopleura exhibits small collapsed cells; but the tissue which fills the folds that dip into the interior consists of much larger cells

The tissue of the albumen is formed of soft-walled parenchyme, which is densely filled with conspicuous starch grains and with fat partly crystallized. Among the prismatic crystals of fat large, thick rhombic or six-sided tables may often be observed. With these are associated grains of albuminoid matter partly crystallized.

In carefully made preparations from the whole nutmeg the structure above described can be made out by care and patience. In the ground spice, however, of these characteristics only the interior parenchyma cells with their starchy contents can be seen when mounted in water, with the alternate use of common and polarized light. The fatty crystals we have not observed, and the fragments of the endopleura or red-brown tissue are characterized only by their color. In chloral hydrate the starch cells and grains are swollen, but the red-brown tissue is much more transparent, sufficiently so, in fact, to reveal any differences between it and any adulterant which might bear a resemblance. There are but few bundles of fibers to be found. The structure as a whole is so simple that the addition of any foreign material can be readily detected.

CHEMICAL COMPOSITION OF NUTMEG.

Analysis of these samples of nutmegs of the following origin have been made:

4512. Whole limed nutmeg, grocers.

4513. Ground limed nutmeg, grocers.

4546. Ground in Baltimore.

Serial No.	Water.	Ash.	Volatile oil.	Fixed oil or fat.	Starch, &c.	Crude fiber.	Albuminoids.	Total.	Nitrogen.
4512.....	6.08	3.27	2.84	34.37	36.98	11.30	5.16	100.00	.83
4513.....	4.19	2.22	3.97	37.30	40.12	6.78	5.42	100.00	.87
4546.....	6.40	3.15	2.90	30.98	41.77	9.55	5.25	100.00	.84

Flückiger states that the volatile oil of nutmegs amounts to between 3 and 8 per cent., and that it is of the nature of a terpene. The fat or fixed oil he places at one-fourth of the weight of the nutmeg, or 25.0 per cent., the principal constituents being starch and albuminoids. Beyond these facts gathered by this authority nothing of value seems to have been published in regard to the proximate percentage composition of nutmegs.

Our results with only three samples are fairly concordant. The volatile oil hardly reached the limit which has been mentioned. It is no doubt a variable quantity. The fixed oil or fat, on the other hand, far exceeds what was expected. Our more perfect methods of extraction may be partly the cause of these results, and it may be also due to a

resinification of the portion of the volatile oil, thus reducing the percentage of this substance and increasing that of the fat.

The amount of starch and allied substances seems fairly constant, while that of albuminoids is rather lower than would be expected. The fiber is also low, but our methods of determining so called fiber are so indefinite that such determinations are of only of comparative value.

Without any actual trials it would seem that determinations of oil and fat, and perhaps of albuminoids and fiber, would serve well to detect foreign substances which would certainly modify in a striking way the normal relations of these proximate principles.

At present nutmegs are almost entirely sold whole and grated in the kitchen. Attempts at adulteration have, therefore, been few in number.

MACE.

The mantle or arillus of the nutmeg, a coat between the outside pericarp and the seed, is known as mace when separated and dried. The coat is not continuous, but is a net-work the form of which is recognizable in the dried spice.

Of its microscopic structure Flückiger and Hanbury say :

The uniform, small-celled angular parenchyme is interrupted by numerous brown oil cells of larger size. The inner part of the tissue contains also thin brown vascular bundles. The cells of the epidermis on either side are colorless, thick walled, longitudinally extended, and covered with a peculiar cuticle of broad, flat ribbon-like cells, which cannot, however, be removed as a continuous film. The parenchyme is loaded with small granules, to which a red color is imparted by Millon's test (solution of mercurous nitrate) and an orange hue by iodine. The granules consequently consist of albuminous matter, and starch is altogether wanting.

This statement has been fully confirmed, and the presence of starch claimed by several writers disproved. Iodine produces a peculiar deep brown color approaching the black-blue given with starch, which, in connection with the granules, has given rise to the statements that starch is present.

In the ground powder of mace all the details of structure described by Flückiger are readily made out, especially in chloral hydrate preparations with polarized light, as the brown vascular bundles, the ribbon-like and epidermal cells all are polarizing substances, while the large mass of granular parenchyma cells are not. The ribbon-like cells are particularly interesting in the varied forms they assume. Adulterations can be readily detected.

CHEMICAL COMPOSITION OF MACE.

Flückiger and Hanbury found in mace 8.2 per cent. of essential oil and 24.5 per cent. of aromatic balsam or resinified aromatic oil but no fat; also 1.4 per cent. of uncrystallizable sugar and 1.8 of mucilage, or a body between that and starch, blued by iodine. The composition of mace they therefore find to be very different from that of nutmeg, contrary to the assertion of Hassall.

Three samples, which we have examined, gave the following results :

4508. Whole mace, guaranteed.
 4509. Ground mace, guaranteed.
 4535. Ground mace, Baltimore.
 4879. Ground mace, Baltimore, low grade.

Analyses of mace.

Serial number.	Water.	Ash.	Volatile oil.	Resin.	Undetermined.	Crude fiber.	Albuminoids.	Total.	Nitrogen.
4508	5.67	4.10	4.04	27.50	41.17	8.93	4.55	100.00	.73
4509	4.86	2.65	8.66	29.08	35.50	4.48	6.13	100.00	.98
4535	10.47	2.20	8.68	23.33	34.68	6.88	5.08	100.00	.81
4879	8.90	3.23	5.39	35.09	28.01	12.20	7.18	100.00	1.15

The percentages of volatile oil agree with the determinations of Flückiger except in the sample ground from the whole nutmeg in our laboratory, which is deficient. The amount probably should always reach 8 per cent. in good nutmegs. The resin found averages rather higher than in Flückiger's specimen, but the presence of so large an amount is certainly very characteristic of this spice, as well of the nutmeg itself, and the presence of adulterants would, without doubt, unless skillfully provided for, be detected by the absence of more than 20 per cent. of this extractive matter.

No qualitative estimation was made of sugar and gum; they were detected qualitatively in small amounts, but from the determinations of fiber and albuminoids it is evident that a large amount of extractive matter not starch is present, and as Flückiger found only about 3 per cent. of substances soluble in alcohol and water and no starch, and as the non-nitrogenous material removed by acids amounts to 35 per cent., it is probably one of the numerous members of the cellulose group, related to gum or starch, which is contained in the parenchyma cells. In the absence of adulterated specimens it is difficult to say what methods of procedure should be taken for their detection; but aside from the microscopical examination, which would readily reveal most adulterants, the chemical characteristics seem to be so marked as to make the presence of any foreign substance evident in the results of analysis.

APPENDIX A.

BIBLIOGRAPHY OF THE LITERATURE OF SPICES AND CONDIMENTS.

The following bibliography consists of a list of such books and papers of any interest as have appeared, to my knowledge, within the last fifteen years. There has seemed to be no necessity for including anything published earlier than 1870, as the progress of investigation has left everything behind which was done before then. Blyth, however, in his work entitled "Foods, their Composition and analyses," gives lists of the ancient literature of the subject, which can be referred to by those who are interested in the historic rather than the analytical aspects of the subject. The works which were published before the date named and which are of interest to the analyst may also be found in a list given by Dr. Battershall in his recent publication.

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A P P E N D I X B.

SOME OF THE LAWS RELATING TO ADULTERATION OF SPICES AND CONDIMENTS.

The following laws are the most valuable and important which have been recently enacted, and are of such character as to make their enforcement possible. There are a few other States having laws which are practically dead letters.

Some portions not relating to the subject of this part of the Bulletin are omitted.*

LEGISLATION.

The New York State general law, of 1881, for the prevention of the adulteration of food and drugs is as follows:

SECTION 1. No person shall, within this State, manufacture, have, offer for sale, or sell any article of food or drugs which is adulterated within the meaning of this act, and any person violating this provision shall be deemed guilty of a misdemeanor, and upon conviction thereof shall be punished by fine not exceeding fifty dollars for the first offense, and not exceeding one hundred dollars for each subsequent offense.

2. The term "food," as used in this act, shall include every article used for food or drink by man. The term "drug," as used in this act, shall include all medicines for internal and external use.

3. An article shall be deemed to be adulterated within the meaning of this act :

a. In the case of drugs.

- (1) If, when sold under or by a name recognized in the United States Pharmacopœia, it differs from the standard of strength, quality, or purity laid down therein.
- (2) If, when sold under or by a name not recognized in the United States Pharmacopœia, but which is found in some other pharmacopœia or other standard work on materia medica, it differs materially from the standard of strength, quality, or purity laid down in such work.
- (3) If its strength or purity fall below the professed standard under which it is sold.

b. In the case of food or drink.

- (1) If any substance or substances has or have been mixed with it so as to reduce or lower or injuriously affect its quality or strength.
- (2) If any inferior or cheaper substance or substances have been substituted wholly or in part for the article.
- (3) If any valuable constituent of the article has been wholly or in part abstracted.
- (4) If it be an imitation of, or be so ld under the name of, another article.
- (5) If it consists wholly or in part of a diseased or decomposed, or putrid or rotten, animal or vegetable substance, whether manufactured or not, or, in the case of milk, if it is the produce of a diseased animal.

* I am indebted to the secretaries of the various States and Dr. Battershall for the material here collected.

- (6) If it be colored, or coated, or polished, or powdered, whereby damage is concealed, or it is made to appear better than it really is, or of greater value.
- (7) If it contain any added poisonous ingredient, or any ingredient which may render such article injurious to the health of the person consuming it: *Provided*, That the State board of health may, with the approval of the governor, from time to time declare certain articles or preparations to be exempt from the provisions of this act: *And provided further*, That the provisions of this act shall not apply to mixtures or compounds recognized as ordinary articles of food, provided that the same are not injurious to health and that the articles are distinctly labeled as a mixture, stating the components of the mixture.

4. It shall be the duty of the State board of health to prepare and publish from time to time lists of the articles, mixtures, or compounds declared to be exempt from the provisions of this act in accordance with the preceding section. The State board of health shall also from time to time fix the limits of variability permissible in any article of food or drug or compound, the standard of which is not established by any national pharmacopœia.

5. The State board of health shall take cognizance of the interests of the public health as it relates to the sale of food and drugs and the adulteration of the same, and make all necessary investigations and inquiries relating thereto. It shall also have the supervision of the appointment of public analysts and chemists, and upon its recommendation whenever it shall deem any such officers incompetent the appointment of any and every such officer shall be revoked and be held to be void and of no effect. Within thirty days after the passage of this act the State board of health shall meet and adopt such measures as may seem necessary to facilitate the enforcement of this act, and prepare rules and regulations with regard to the proper methods of collecting and examining articles of food or drugs, and for the appointment of the necessary inspectors and analysts; and the State board of health shall be authorized to expend, in addition to all sums already appropriated for said board, an amount not exceeding ten thousand dollars for the purpose of carrying out the provisions of this act. And the sum of ten thousand dollars is hereby appropriated, out of any moneys in the treasury not otherwise appropriated, for the purposes in this section provided.

6. Every person selling or offering or exposing any article of food or drugs for sale, or delivering any article to purchasers, shall be bound to serve or supply any public analyst or other agent of the State or local board of health appointed under this act, who shall apply to him for that purpose, and on his tendering the value of the same, with a sample sufficient for the purpose of analysis of any article which is included in this act, and which is in the possession of the person selling, under a penalty not exceeding \$50 for a first offense and \$100 for a second and subsequent offense.

7. Any violation of the provisions of this act shall be treated and punished as a misdemeanor; and whoever shall impede, obstruct, hinder, or otherwise prevent any analyst, inspector, or prosecuting officer in the performance of his duty shall be guilty of a misdemeanor, and shall be liable to indictment and punishment therefor.

8. Any acts or parts of acts inconsistent with the provisions of this act are hereby repealed.

9. All the regulations and declarations of the State board of health made under this act from time to time, and promulgated, shall be printed in the statutes at large.

10. This act shall take effect at the expiration of ninety days after it shall become a law.

Amendment of April 29, 1885.

SECTION 1. The title of chapter 407 of the laws of 1881, entitled "An act to prevent the adulteration of food and drugs," is hereby amended to read as follows: "An act

to prevent the adulteration of food, drugs, and spirituous, fermented, or malt liquors in the State of New York."

* * * * *

3. Section 2 is hereby amended to read as follows :

"2. The term food as used in this act shall include every article of food or drink by man, including teas, coffees, and spirituous, fermented, and malt liquors. The term drug as used in this act shall include all medicines for internal or external use."

* * * * *

5. Section 5 is hereby amended to read as follows :

"5. The State board of health shall take cognizance of the interests of the public health as relates to the sale of food, drugs, spirituous, fermented, and malt liquors, and the adulteration thereof, and make all necessary inquiries relating thereto. It shall have the supervision of the appointment of public analysts and chemists, and upon its recommendation, whenever it shall deem any such officers incompetent, the appointment of any and every such officer shall be revoked and be held to be void and of no effect. Within thirty days after the passage of this act, and from time to time thereafter as it may deem expedient, the said board of health shall meet and adopt such measures, not provided for by this act, as may seem necessary to facilitate the enforcement of this act, and for the purpose of making an examination or analysis of spirituous, fermented, or malt liquors sold or exposed for sale in any store or place of business not herein otherwise provided for, and prepare rules and regulations with regard to the proper methods of collecting and examining articles of food, drugs, spirituous, fermented, or malt liquors, and for the appointment of the necessary inspectors and analysts. The said board shall at least once in the calendar year cause samples to be procured, in public market or otherwise, of the spirituous, fermented, or malt liquors distilled, brewed, manufactured, or offered for sale in each and every brewery or distillery located in this State, and a test, sample, or analysis thereof to be made by a chemist or analyst duly appointed by said board of health. The samples shall be kept in vessels and in a condition necessary and adequate to obtain a proper test and analysis of the liquors contained therein. The vessels containing such samples shall be properly labeled and numbered by the secretary of said board of health, who shall also prepare and keep an accurate and proper list of the names of the distillers, brewers, or vendors, and opposite each name shall appear the number which is written or printed upon the label attached to the vessel containing the sample of the liquor manufactured, brewed, distilled, or sold. Such lists, numbers, and labels shall be exclusively for the information of the said board of health, and shall not be disclosed or published unless upon discovery of some deleterious substance prior to the completion of the analysis, except when required in evidence in a court of justice. The samples when listed and numbered shall be delivered to the chemist, analyst, or other officer of said board of health, and shall be designated and known to such chemist, analyst, or officer only by its number, and by no other mark or designation. The result of the analysis or investigation shall thereupon, and within a convenient time, be reported by the officer conducting the same to the secretary of said State board of health, setting forth explicitly the nature of any deleterious substance, compound, or adulteration which may be detrimental to public health and which has been found upon analysis in such samples, and stating the number of the samples in which said substance was found. Upon such examination or analysis the brewer, distiller, or vendor in whose sample of spirituous, fermented, or malt liquor such deleterious substances, compounds, or adulterations shall be found, shall be deemed to have violated the provisions of this act, and shall be punishable as prescribed in section 7 of this act."

* * * * *

7. Section 7 of said chapter 407 of the laws of 1881 is hereby amended to read as follows :

"7. Upon discovering that any person has violated any of the provisions of this act, the State board of health shall immediately communicate the facts to the district at-

torney of the county in which the person accused of such violation resides or carries on business, and the said district attorney, upon receiving such communication or notification, shall forthwith commence proceeding for indictment and trial of the accused as prescribed by law in cases of misdemeanor."

8. The State board of health shall be authorized to expend, in addition to the sums already appropriated for said board, an amount not exceeding \$3,000, for the purpose of carrying out the provisions of this act, in relation to spirituous, fermented, or malt liquors. And the sum of \$3,000 is hereby appropriated out of any moneys in the treasury not otherwise appropriated and expended for the purposes of this act.

9. This act shall take effect immediately.

The New Jersey general law is the same as that of New York.

Following are the rules of the New Jersey board of health for its inspectors and analysts:

DUTIES OF INSPECTORS.

1. The inspector is to buy samples of food or drugs, and to seal each sample in the presence of a witness.

2. The inspector must affix to each sample a label bearing a number, his initials, and the date of purchase.

3. Under no circumstance is the inspector to inform the analyst as to the source of the sample before the analysis shall have been completed and formally reported to the president or secretary of the state board of health.

4. Inspectors are to keep a record of each sample as follows:

(1) Number of sample.

(2) Date and time of purchase.

(3) Name of witness to sealing.

(4) Name and address of seller.

(5) Name and address of producer, manufacturer, or wholesaler, when known, with marks on original package.

(6) Name of analyst and date of sending.

(7) How sent to analyst.

5. If the seller desires a portion of the sample, the inspector is to deliver it under seal. The duplicate sample left with seller should have a label containing the same marks as are affixed to the portion taken by the inspector.

6. The inspector is to deliver the sample to the analyst, taking his receipt for the same, or he may send it by registered mail, express, or special messenger.

DUTIES OF THE ANALYSTS.

1. The analyst is to analyze the samples immediately upon receipt thereof.

2. Samples, with the exception of milk and similar perishable articles, are to be divided by the analyst and a portion sealed up, and a copy of the original label affixed. These duplicates are to be sent to the secretary of the state board of health at the end of each month, and to be retained by him until demanded for another analysis, as provided for in section 3 of these rules.

3. Should the result obtained by any analyst be disputed in any case, an appeal may be made to the State board of health, through its secretary, by the defendant or person selling the sample, or his attorney, and said secretary shall then require another member of the committee of public analysts to repeat the analysis, using the duplicate sample for such purpose. But when an appeal shall be made, a sum of money sufficient to cover the expenses of the second analysis shall be deposited with the president of the state board of health, which sum shall be paid over to the analyst designated by the president and secretary of the board to perform the second analysis, in case the analysis shall be found to agree with the first in all essential particulars.

4. In the case of all articles having a standard of purity fixed by any of the laws of the State, the certificate of the analyst should show the relation of the article in question to that standard.

5. Where standards of strength, purity, or quality are not fixed by law, the committee of analysts shall present to the State board of health such standard as in their judgment should be fixed.

6. Each analyst should keep a record book, in which should be entered notes, as follows:

- (1) From whom the sample is received.
- (2) Date, time, and manner in which the sample was received.
- (3) Marks on package, sealed or not.
- (4) Results of analysis in detail.

This record should be produced at each meeting of the committee.

7. At the completion of the analysis a certificate in the form given below should be forwarded to the person from whom the sample was received, and a duplicate copy sent to the State board of health.

CERTIFICATE.

To whom it may concern:

I, ———, a member of the committee of public analysts, appointed by the State board of health of New Jersey under provisions of an act entitled "An act to prevent the adulteration of food and drugs," approved March 25, 1881, do hereby certify that I received from ———, on the ——— day of ———, 188—, a sample of ———, sealed as required by the rules of said board, and bearing the following words, to wit:—

I carefully mixed said samples and have analyzed the same, and hereby certify and declare the results of my analyses to be as follows: ———.

[Signature.] ——— ———.

EXCEPTIONS.

The following exceptions are adopted:

Mustard.—Compounds of mustard with rice flour, starch, or flour may be sold if each package is marked "Compound Mustard," and if not more than 25 per cent. of such substance is added to the mustard.

Coffee.—Compounds of coffee with chicory, rye, wheat, or other cereals, may be sold if the package is marked "A Mixture," and if the label states the per cent. of coffee contained in said mixture.

Oleomargarine and other imitation dairy products may be sold if each package is marked with the name of the substance, and in all respects fulfils the terms of the special law as to these.

Sirups.—When mixed with glucose, sirup may be sold if the package is marked "A Mixture."

The following are the statutes of the State of Massachusetts relating to the adulteration of food and drugs:

GENERAL LAWS RELATING TO ADULTERATION.

FOOD AND DRUGS.

SECTION 1. No person shall, within this Commonwealth, manufacture for sale, offer for sale, or sell any drug or article of food which is adulterated within the meaning of this act.

2. The term "drug" as used in this act shall include all medicines for internal or external use, antiseptics, disinfectants, and cosmetics. The term "food" as used herein shall include all articles used for food or drink by man.

3. An article shall be deemed to be adulterated within the meaning of this act—

(a) *In the case of drugs.*—(1) If, when sold under or by a name recognized in the United States Pharmacopœia, it differs from the standard of strength, quality, or

purity laid down therein, unless the order calls for an article inferior to such standard, or unless such difference is made known or so appears to the purchaser at the time of such sale; (2) if, when sold under or by a name not recognized in the United States Pharmacopœia, but which is found in some other pharmacopœia, or other standard work on *materia medica*, it differs materially from the standard of strength, quality, or purity laid down in such work; (3) if its strength or purity falls below the professed standard under which it is sold.

(b) *In the case of food.*—(1) If any substance or substances have been mixed with it so as to reduce, or lower, or injuriously affect its quality or strength; (2) if any inferior or cheaper substance or substances have been substituted wholly or in part for it; (3) if any valuable constituent has been wholly or in part abstracted from it; (4) if it is an imitation of or is sold under the name of another article; (5) if it consists wholly or in part of a diseased, decomposed, putrid, or rotten animal or vegetable substance, whether manufactured or not, or in the case of milk, if it is the produce of a diseased animal; (6) if it is colored, coated, polished, or powdered, whereby damage is concealed, or if it is made to appear better or of greater value than it really is; (7) if it contains any added or poisonous ingredient, or any ingredient which may render it injurious to the health of a person consuming it.

4. The provisions of this act shall not apply to mixtures or compounds recognized as ordinary articles of food or drinks, provided that the same are not injurious to health and are distinctly labeled as mixtures or compounds. And no prosecutions shall at any time be maintained under the said act concerning any drug the standard of strength or purity whereof has been raised since the issue of the last edition of the United States Pharmacopœia, unless and until such change of standard has been published throughout the Commonwealth.

5. The State board of health, lunacy, and charity shall take cognizance of the interests of the public health relating to the sale of drugs and food and the adulteration of the same, and shall make all necessary investigations and inquiries in reference thereto, and for these purposes may appoint inspectors, analysts, and chemists, who shall be subject to its supervision and removal.

Within thirty days after the passage of this act the said board shall adopt such measures as it may deem necessary to facilitate the enforcement hereof, and shall prepare rules and regulations with regard to the proper methods of collecting and examining drugs and articles of food. Said board may expend annually an amount not exceeding ten thousand dollars for the purpose of carrying out the provisions of this act: *Provided, however,* That not less than three-fifths of the said amount shall be annually expended for the enforcement of the laws against the adulteration of milk and milk products.

6. Every person offering or exposing for sale, or delivering to a purchaser, any drug or article of food included in the provisions of this act, shall furnish to any analyst or other officer or agent appointed hereunder, who shall apply to him for the purpose and shall tender him the value of the same, a sample sufficient for the purpose of the analysis of any such drug or article of food which is in his possession.

7. Whoever hinders, obstructs, or in any way interferes with any inspector, analyst, or other officer appointed hereunder, in the performance of his duty, and whoever violates any of the provisions of this act, shall be punished by a fine not exceeding fifty dollars for the first offense, and not exceeding one hundred dollars for each subsequent offense.

8. The State board of health, lunacy, and charity shall report annually to the legislature the number of prosecutions made under said chapter, and an itemized account of all money expended in carrying out the provisions thereof.

9. An inspector appointed under the provisions of said chapter two hundred and sixty-three of the acts of the year eighteen hundred and eighty-two shall have the same powers and authority conferred upon a city or town inspector by section two of chapter fifty-seven of the public statutes.

10. Nothing contained in chapter two hundred and sixty-three of the acts of the year eighteen hundred and eighty-two shall be in any way construed as repealing or amending anything contained in chapter fifty-seven of the public statutes.

11. Before commencing the analysis of any sample, the person making the same shall reserve a portion which shall be sealed; and in case of a complaint against any person the reserved portion of the sample alleged to be adulterated shall upon application be delivered to the defendant or his attorney.

12. Whoever knowingly sells any kind of diseased, corrupted, or unwholesome provisions, whether for meat or drink, without making the same fully known to the buyer, shall be punished by imprisonment in the jail not exceeding six months, or by fine not exceeding two hundred dollars.

13. Whoever fraudulently adulterates, for the purpose of sale, bread or any other substance intended for food, with any substance injurious to health, or knowingly barter, gives away, sells, or has in possession with intent to sell, any substance intended for food, which has been adulterated with any substance injurious to health, shall be punished by imprisonment in the jail not exceeding one year, or by fine not exceeding three hundred dollars; and the articles so adulterated shall be forfeited and destroyed under the direction of the court.

14. Whoever adulterates, for the purpose of sale, any liquors used or intended for drink, with Indian cockle, vitriol, grains of paradise, opium, alum, capsicum, copperas, laurel-water, logwood, Brazil wood, cochineal, sugar of lead, or any other substance which is poisonous or injurious to health, and whoever knowingly sells any such liquor so adulterated, shall be punished by imprisonment in the State prison not exceeding three years; and the articles so adulterated shall be forfeited.

15. Whoever fraudulently adulterates, for the purpose of sale, any drug or medicine, or sells any fraudulently adulterated drug or medicine, knowing the same to be adulterated, shall be punished by imprisonment in the jail not exceeding one year or by fine not exceeding four hundred dollars; and such adulterated drugs and medicines shall be forfeited and destroyed under the direction of the court.

16. Whoever sells arsenic, strychnine, corrosive sublimate, or prussic acid, without the written prescription of a physician, shall keep a record of the date of such sale, the name of the article, the amount thereof sold, and the name of the person or persons to whom delivered; and for each neglect shall forfeit a sum not exceeding fifty dollars. Whoever purchases deadly poisons as aforesaid, and gives a false or fictitious name to the vendor, shall be punished by fine not exceeding fifty dollars.

CHAP. 171.—AN ACT concerning the adulteration of food and drugs.

Be it enacted, etc., as follows :

Section two of chapter two hundred and sixty-three of the acts of the year eighteen hundred and eighty-two is hereby amended so as to read as follows: The term "drug" as used in this act shall include all medicines for internal or external use, antiseptics, disinfectants, and cosmetics. The term "food" as used herein shall include confectionery, condiments, and all articles used for food or drink by man.—Approved April 29, 1886.

RULES AND REGULATIONS OF THE STATE BOARD OF HEALTH, LUNACY, AND CHARITY OF MASSACHUSETTS RELATIVE TO THE INSPECTION AND ANALYSIS OF FOOD AND DRUGS.

1. The state board of health, lunacy, and charity shall appoint analysts and inspectors, as provided in section 5 of chapter 263, acts of 1882.

2. It shall be the duty of the inspectors to procure samples of drugs and articles of food at such times and places as the health officer shall direct, in the manner provided in section 6 of chapter 263 of the acts of 1882, and in section 3 of chapter 289 of the acts of 1884, and in all acts amendatory of said provisions.

3. Under the direction of the health officer, one of the inspectors shall, for the identification of samples, affix a number to each sample of food or drugs obtained by him, beginning with number one, and taking every alternate or odd number thereafter, without limit; and the other inspector shall use and affix each alternate or even number, beginning with number two, and following such form of numbering, without limit, also, as far as may be directed. Under no circumstances shall an inspector convey any information to an analyst as to the source from which any sample was obtained.

4. The inspectors shall keep records of each sample, each record to include the following items:

- (a) The inspector's number.
- (b) The date of purchase or receipt of sample.
- (c) The character of the sample.
- (d) The name of the vender.
- (e) The name of the city or town and street and number where the sample is obtained, and in the case of a licensed milk peddler, the number of his license.
- (f) As far as possible, the names of manufacturers, producers, or wholesalers, with marks, brands, or labels stamped or printed upon goods.

5. It shall be the duty of the analysts so appointed to determine, under the direction of the health officer, by proper examination and analysis, whether articles of food and drugs, manufactured for sale, offered for sale, or sold within this Commonwealth, are adulterated within the meaning of chapter 263 of the acts and resolves passed by the general court of Massachusetts in 1882, and all acts amendatory thereof, adulteration being defined as follows, viz:

In the case of drugs, (1) If sold under or by a name recognized in the United States Pharmacopœia, it differs from the standard of strength, quality, or purity laid down therein, unless the order calls for an article inferior to such standard, or unless such difference is made known or so appears to the purchaser at the time of such sale. (2) If, when sold under or by a name not recognized in the United States Pharmacopœia, but which is found in some other pharmacopœia or standard work on materia medica, it differs materially from the standard of strength, quality, or purity laid down in such work. (3) If its strength or purity falls below the professed standard under which it is sold.

In the case of food, (1) If any substance or substances have been mixed with it, so as to reduce or lower or injuriously affect its quality or strength. (2) If any inferior or cheaper substance or substances have been substituted, wholly or in part, for it. (3) If any valuable constituent has been wholly or in part abstracted from it. (4) If it is an imitation of or is sold under the name of another article. (5) If it consists wholly or in part of a diseased, decomposed, putrid, or rotten animal or vegetable substance, whether manufactured or not, or in the case of milk, if it is the produce of a diseased animal. (6) If it is colored, coated, polished, or powdered, whereby damage is concealed, or if it is made to appear of better or of greater value than it really is. (7) If it contains any added poisonous ingredient, or any ingredient which may render it injurious to the health of the person consuming it.

6. It shall also be the duty of the analysts to receive such specimens of food and drugs for analysis as may be delivered to them by the health officer, or by the inspectors, and to examine the same. To avoid, as far as possible, all suggestion or danger of specimens having been tampered with, each analyst shall keep each specimen in his possession in a suitable and secure place, labeled in such a manner as to prevent any person from having access to the same, without the knowledge and presence of the analyst.

Analyses of perishable articles should be made promptly after they are received.

7. An analyst shall give no information, under any circumstances, regarding the result of any analysis to any person except to the health officer of the board, prior to any trial in court in reference to such analysis.

The analysts shall carefully avoid any error regarding the inspector's number attached to each sample, and shall report the results of their work in detail to the health officer.

In the case of all articles having a numerical standard provided by statute, the result of the analysis should show their relation to such standard.

8. Before beginning the analysis of any sample, the analyst shall reserve a portion, which shall be sealed, and in the event of finding the portion analyzed to be adulterated, he shall preserve the sealed portion, so that in case of a complaint against any person the last-named portion may, on application, be delivered by the health officer to the defendant or to his attorney.

9. Each analyst shall present to the health officer on the Thursday before the first Saturday of each month, a summary of the analyses made by him during the previous month.

Each analyst shall also present, on or before the first of January of each year, an annual report of the work done for the year ending on the 30th of September preceding.

10. The health officer shall have charge of the reports of analyses, and shall cause cases founded on such reports to be submitted to the courts for prosecution.

In each case of a retailer, and of every dealer not a manufacturer or producer, he may, if the party has not been previously complained of in court, issue a notice or warning of any violation of the law relative to the adulteration of food and drugs, and of the offender's liability to prosecution on a repetition of the sale.

11. Should the result obtained by any analyst be questioned in any given case, another analyst shall repeat the analysis, unless otherwise instructed by the board, provided a sufficient sum to meet the expense of the analysis be deposited with the health officer by any interested party feeling aggrieved, which sum will not be returned unless the second analysis fails to confirm the first in essential particulars.

12. Any appeal from the decision of an analyst shall be filed with the health officer, who shall report it, and any matter in controversy, to the board, giving his judgment thereon, and the board shall supervise and control the action of its officers, in executing the law.

13. Where standards of strength, quality, or purity are not fixed by the act, the analysts shall present to the health officer such standard as in their judgment should be fixed, and the health officer shall report the same to the board for its action. The standards set by the British Society of Public Analysts will be followed as nearly as practicable, until otherwise ordered.

14. Whenever a drug or preparation not described in a National Pharmacopœia or other standard work on *materia medica*, shall be manufactured, offered for sale, or used in this State, the standard of such drug, and the standard and proportion of the ingredients of such preparation, and the range of variability from such standard or standards shall be ascertained by the analysts, who shall report the same through the health officer to the board.

15. The analysts shall occupy such time in the performance of their respective duties as a reasonable compliance with the terms of the statute shall require, and shall be present one hour of each day at such time of the day and at such place as shall be designated by the committee on health of the board, to meet the convenience of interested parties and the public.

16. The compensation of the analyst of articles of food shall be at the rate of \$1,500 per annum, and that of the analyst of drugs shall be at the rate of \$1,000.

That of the analyst of milk for the ten eastern counties of the Commonwealth shall be at the rate of \$800 per annum, and that of the analyst of the four western counties shall be at the rate of \$500 per annum.

The compensation of each inspector shall be at the rate of \$1,000 per annum.

The laws of Michigan are as follows:

ADULTERATION OF FOODS, DRINKS, DRUGS, OR MEDICINES.

208. (7727.) SEC. 2. If any person shall fraudulently adulterate, for the purpose of sale, any substance intended for food, or any wine, spirits, malt liquor, or other liquor intended for drinking, he shall be punished by imprisonment in the county jail not more than one year, or by fine not exceeding three hundred dollars, and the article so adulterated shall be forfeited and destroyed.—§9317.

209. (7728.) SEC. 3. If any person shall fraudulently adulterate, for the purpose of sale, any drug or medicine, in such manner as to render the same injurious to health, he shall be punished by imprisonment in the county jail not more than one year, or by fine not exceeding four hundred dollars, and such adulterated drugs and medicines shall be forfeited and destroyed.—§9318.

ADULTERATION OF FOODS, DRINKS, AND MEDICINES. AND SALE THEREOF WHEN ADULTERATED.

Act No. 254, laws of 1881, entitled "An Act to prevent and punish the adulteration of articles of food, drink, and medicine, and the sale thereof when adulterated."

210. SECTION 1. *The People of the State of Michigan enact*, That no person shall mix, color, stain, or powder, or order or permit any other person to mix, color, stain, or powder any article of food with any ingredient or material so as to render the article injurious to health, with the intent that the same may be sold; and no person shall knowingly sell or offer for sale any such article so mixed, colored, stained, or powdered.—§9324.

211. SEC. 2. No person shall, except for the purpose of compounding in the necessary preparation of medicine, mix, color, stain, or powder, or order or permit any other person to mix, color, stain, or powder, any drug or medicine with any ingredient or ingredients or materials so as to affect injuriously the quality or potency of such drug or medicine, with intent to sell the same, or shall sell or offer for sale any such drug or medicine so mixed, colored, stained, or powdered.—§9325.

212. SEC. 3. No person shall mix, color, stain, or powder any article of food, drink, or medicine, or any article which enters into the composition of food, drink, or medicine, with any other ingredient or material, whether injurious to health or not, for the purpose of gain or profit, or sell or offer the same for sale, or order or permit any other person to sell or offer for sale any article so mixed, colored, stained, and powdered, unless the same be so manufactured, used, or sold, or offered for sale under its true and appropriate name, and notice that the same is mixed or impure is marked, printed, or stamped upon each package, roll, parcel, or vessel containing the same, so as to be and remain at all times readily visible, or unless the person purchasing the same is fully informed by the seller of the true name and ingredients (if other than such as are known by the common name thereof) of such article of food, drink, or medicine at the time of making sale thereof or offering to sell the same.—§9326.

213. SEC. 4. No person shall mix any glucose or grape sugar with syrup, honey, or sugar, intended for human food, or any oleomargarine, suine, beef fat, lard, or any other foreign substance, with any butter or cheese intended for human food, or shall mix or mingle any glucose or grape sugar or oleomargarine with any article of food, without distinctly marking, stamping, or labeling the article, or the package containing the same, with the true and appropriate name of such article, and the percentage in which glucose or grape sugar, oleomargarine, or suine, enter into its composition; nor shall any person sell, or offer for sale, or order or permit to be sold, or offered for sale, any such food into the composition of which glucose, or grape sugar, or oleomargarine, or suine has entered, without at the same time informing the buyer of the fact, and the proportions in which such glucose or grape sugar, oleomargarine, or suine has entered into its composition.—§9327.

214. SEC. 5. Any person convicted of violating any provision of any of the foregoing sections of this act shall be fined not more than fifty dollars or imprisoned in the county jail not exceeding three months.—§9328.

215. SEC. 6. It is hereby made the duty of the prosecuting attorneys of this State to appear for the people and to attend to the prosecution of all complaints under this act in all the courts in their respective counties.—§9329.

216. SEC. 7. All acts and parts of acts inconsistent with the provisions of this act are hereby repealed.—§9330.

The Canadian law is as follows, in addition to which there is an elaborate act respecting the inspection of staple articles of Canadian produce:

CHAPTER 107.—An Act respecting the adulteration of food, drugs, and agricultural fertilizers.

Her Majesty, by and with the advice and consent of the Senate and House of Commons of Canada, enacts as follows:

SHORT TITLE.

1. This act may be cited as "*The adulteration act.*"—48-49 V., c. 67, s. 1.

INTERPRETATION.

2. In this act, unless the context otherwise requires—

(a) The expression "food" includes every article used for food or drink by man or by cattle.

(b) The expression "drug" includes all medicines for internal or external use for man or for cattle.

(c) The expression "agricultural fertilizer" means and includes every substance imported, manufactured, prepared, or disposed of for fertilizing or manuring purposes which is sold at more than ten dollars per ton and which contains phosphoric acid or ammonia or its equivalent of nitrogen.

(d) The expression "officer" means any officer of inland revenue or any person authorized under this act or "*the fertilizers act*" to procure samples of articles of food, drugs, or agricultural fertilizers and to submit them for analysis.

(e) Food shall be deemed to be "adulterated" within the meaning of this act—

(1) If any substance has been mixed with it so as to reduce or lower or injuriously affect its quality or strength.

(2) If any inferior or cheaper substance has been substituted, wholly or in part, for the article.

(3) If any valuable constituent of the article has been wholly or in part abstracted.

(4) If it is an imitation of, or is sold under the name of, another article.

(5) If it consists wholly or in part of a diseased or decomposed or putrid or rotten animal or vegetable substance, whether manufactured or not, or, in the case of milk or butter, if it is the produce of a diseased animal or of an animal fed upon unwholesome food.

(6) If it contains any added poisonous ingredient or any ingredient which may render such an article injurious to the health of a person consuming it.

(f) Every drug shall be deemed to be "adulterated" within the meaning of this act—

(1) If, when sold or offered or exposed for sale under or by a name recognized in the British or United States pharmacopœia, it differs from the standard of strength, quality, or purity laid down therein.

(2) If, when sold or offered or exposed for sale under or by a name not recognized in the British or United States pharmacopœia, but which is found in some other generally recognized pharmacopœia or other standard work on *materia medica*, it differs from the standard of strength, quality, or purity laid down in such work.

(3) If its strength or purity falls below the professed standard under which it is sold or offered or exposed for sale.

(g) Provided, that the foregoing definitions as to the adulteration of food and drugs shall not apply—

(1) If any matter or ingredient not injurious to health has been added to the food or drug because the same is required for the production or preparation thereof as an article of commerce, in a state fit for carriage or consumption, and not fraudulently to increase the bulk, weight, or measure of the food or drug or to conceal the inferior quality thereof, if such articles are distinctly labeled as a mixture, in conspicuous characters, forming an inseparable part of the general label, which shall also bear the name and address of the manufacturer.

(2) If the food or drug is a proprietary medicine, or is the subject of a patent in force, and is supplied in the state required by the specification of the patent.

(3) If the food or drug is unavoidably mixed with some extraneous matter in the process of collection or preparation.

(4) If any articles of food not injurious to the health of the person consuming the same are mixed together and sold or offered for sale as a compound, and if such articles are distinctly labeled as a mixture, in conspicuous characters, forming an inseparable part of the general label, which shall also bear the name and address of the manufacturer.

(h) Every agricultural fertilizer shall be deemed to be “adulterated” within the meaning of this act if, when sold, offered, or exposed for sale, the chemical analysis thereof shows a deficiency of more than one per cent. of any of the chemical substances, the percentages whereof are required to be specified in the certificate, by “the fertilizers act” required to be affixed to each barrel, box, sack, or package containing the same, or (if the agricultural fertilizer is in bulk) to be produced to the inspector; or if it contains less than the minimum percentage of such substances required by the said act to be contained in such fertilizer. 48-49 V., c. 67, s. 2.

ANALYSIS.

3. The governor in council may appoint one or more persons possessing competent medical, chemical, and microscopical knowledge as analysts of food, drugs, and agricultural fertilizers purchased, sold, or exposed or offered for sale within such territorial limits as are assigned to each of them respectively, and may also select from among the aforesaid analysts so appointed, or may appoint, in addition thereto, a chief analyst, who shall be attached to the staff of the department of inland revenue at Ottawa.

(2) No analyst shall be appointed until he has undergone an examination before a special examining board appointed by the governor in council, and until he has obtained from such board a certificate setting forth that he is duly qualified to perform the duties attached to the office of analyst. 48-49 V., c. 67, s. 3; 49 V., c. 41, s. 1.

4. The governor in council may cause such remuneration to be paid to such chief analyst and to such analysts as he deems proper, and such remuneration, whether by fees, or salary, or partly in one way, and partly in the other, may be paid to them out of any sums voted by Parliament for the purposes of this act. 48-49 V., c. 67, s. 4.

5. The officers of inland revenue, the inspectors and deputy inspectors of weights and measures, and the inspectors and deputy inspectors acting under “the general inspection act,” or any of them, shall, when required so to do by any regulation made in that behalf by the minister of inland revenue, procure and submit samples of food, drugs, or agricultural fertilizers suspected to be adulterated, to be analyzed by the analysts appointed under this act. 48-49 V., c. 67, s. 5.

6. The council of any city, town, county, or village may appoint one or more inspectors of food, drugs, and agricultural fertilizers; and such inspectors shall, for the purposes of this act, have all the powers by this act vested in officers of inland revenue; and any such inspector may require any public analyst to analyze any sam-

ples of food, drugs, or agricultural fertilizers collected by him, if such samples have been collected in accordance with the requirements of this act.

(2) The said analyst shall, upon tender of the fees fixed for the analysis of such class of articles by the governor in council, forthwith analyze the same, and give the inspector a certificate of such analysis.

(3) Such inspector may prosecute any person manufacturing, selling, or offering or exposing for sale within the city, county, town, or village for which he is appointed inspector, any article of food, drug, or agricultural fertilizer which has been certified by any public analyst to have been adulterated within the meaning of this act.

(4) Notwithstanding any other provision of this act in respect of the disposition of penalties, all penalties imposed and recovered at the suit of any such inspector shall be paid into the revenue of the city, county, town, or village by the council of which such inspector was appointed, and may be distributed in such manner as the council of such city, county, town, or village by by-law directs. 48-49 V., c. 67, s. 6.

7. Any officer may procure samples of food, drugs, or agricultural fertilizers which have not been declared exempt from the provisions of this act, from any person who has such articles in his possession for the purpose of sale, or who sells or exposes the same for sale; and he may procure such samples either by purchasing the same or by requiring the person in whose possession they are to show him and allow him to inspect all such articles in his possession, and the place or places in which such articles are stored, and to give him samples of such articles, on payment or tender of the value of such samples. 48-49 V., c. 67, s. 7.

8. If the person who has such articles in his possession, or his agent or servant, refuses or fails to admit the officer, or refuses or omits to show all or any of the said articles in his possession, or the place in which any such articles are stored, or to permit the officers to inspect the same, or to give any samples thereof, or to furnish the officer with such light or assistance as he requires, when required so to do in pursuance of this act, he shall be liable to the same penalty as if he knowingly sold or exposed for sale adulterated articles knowing them to be adulterated. 48-49 V., c. 67, s. 8.

9. The officer purchasing any article with the intention of submitting the same to be analyzed, shall, after the purchase has been completed, forthwith notify the seller or his agent selling the article of his intention to have the same analyzed by the public analyst, and shall, except in specific cases, respecting which provision is made by the governor in council, divide the article into three parts—to be then and there separated, and each part to be marked and sealed up, as its nature permits—and shall deliver one of the parts to the seller or his agent, if required by him so to do.

(2) He shall transmit another of such parts to the minister of inland revenue for submission to the chief analyst in case of appeal, and shall submit the remaining part to the analyst for the district within which the samples were taken, unless otherwise directed by the minister of inland revenue. 48-49 V., c. 67, s. 9.

10. The person from whom any sample is obtained under this act may require the officer obtaining it to annex to the vessel or package containing the part of the sample which he is hereby required to transmit to the minister of inland revenue the name and address of such person, and to secure, with a seal or seals belonging to him, the vessel or package containing such part of the sample, and the address annexed thereto, in such manner that the vessel or package cannot be opened, or the name and address taken off, without breaking such seals; and the certificate of the chief analyst shall state the name and address of the person from whom the said sample was obtained, that the vessel or package was not open, and that the seals, securing to the vessel or package the name and address of such person, were not broken until such time as he opened the vessel or package for the purpose of making his analysis; and in such case no certificate shall be receivable in evidence, unless there is contained therein such statement as above, or a statement to the like effect. 48-49 V., c. 67, s. 10.

11. When the officer has, by either of the means aforesaid, procured samples of the articles to be analyzed, he shall cause the same to be analyzed by one of the analysts appointed under this act, and if it appears to the analyst that the sample is adulterated within the meaning of this act, he shall certify such fact, stating in such certificate, in the case of an article of food or a drug, whether such adulteration is of a nature injurious to the health of the person consuming the same; and the certificate so given shall be received as evidence in any proceedings taken against any person in pursuance of this act, subject to the right of any person against whom proceedings are taken to require the attendance of the analyst, for the purpose of cross-examination. 48-49 V., c. 67, s. 11.

12. If the vendor of the article respecting which such certificate is given deems himself aggrieved thereby, he may, within forty-eight hours of the receipt of the first notification of the intention of the officer or other purchaser to take proceedings against him (whether such notification is given by the purchaser or by the ordinary process of law), notify the said officer or purchaser in writing that he intends to appeal from the decision of the analyst to the judgment of the chief analyst: and in such case the officer or purchaser shall transmit such notification to the chief analyst, and the chief analyst shall, with all convenient speed, analyze the part of the sample transmitted to the minister of inland revenue for that purpose, and shall report thereon to the said minister; and the decision of such chief analyst shall be final, and his certificate thereof shall have the same effect as the certificate of the analyst in the next preceding section mentioned. 48-49 V., c. 67, s. 12.

13. Every analyst appointed under this act shall report quarterly to the minister of inland revenue the number of articles of food, drugs, and agricultural fertilizers analyzed by him under this act during the preceding quarter, and shall specify the nature and kind of adulterations detected in such articles of food, drugs, and agricultural fertilizers; and all such reports, or a synopsis of them, and the names of the vendors or persons from whom obtained, and of the manufacturers when known, shall be printed and laid before Parliament as an appendix to the annual report of the said minister. 48-49 V., c. 67, s. 13.

ADULTERATION.

14. No person shall manufacture, expose or offer for sale, or sell any food, drug, or agricultural fertilizer which is adulterated within the meaning of this act. 48-49 V., c. 67, s. 14.

15. If milk is sold, or offered or exposed for sale, after any valuable constituent of the article has been abstracted therefrom, or if water has been added thereto, or if it is the product of a diseased animal fed upon unwholesome food, it shall be deemed to have been adulterated in a manner injurious to health, and such sale, offer, or exposure for sale shall render the vendor liable to the penalty hereinafter provided in respect to the sale of adulterated food; except that skimmed milk may be sold as such if contained in cans bearing upon their exterior, within twelve inches of the tops of such vessels, the word "skimmed" in letters of not less than two inches in length, and served in measures also similarly marked; but any person supplying such skimmed milk, unless such quality of milk has been asked for by the purchaser, shall not be entitled to plead the provisions of this section as a defense to or in extenuation of any violation of this act:

(2) Nothing in this section shall be interpreted to permit or warrant the admixture of water with milk, or any other process than the removal of cream by skimming. 48-49 V., c. 67, s. 15.

16. Vinegar sold, or offered or exposed for sale, shall be deemed to be adulterated in a manner injurious to health if any mineral acid has been added thereto, or if it contains any soluble salt having copper or lead as a base thereof—whether such mineral acid or salt is added either during the process of manufacture or subsequently. 48-49 V., c. 67, s. 16.

17. Alcoholic, fermented, or other potable liquors sold, or offered or exposed for sale, shall be deemed to have been adulterated in a manner injurious to health if they are found to contain any of the articles mentioned in the schedule of this act, or any article hereafter added to such schedule by the governor in council. 48-49 V., c. 67, s. 17.

18. The governor in council may from time to time declare certain articles or preparations exempt in whole or in part from the provisions of this act, and may add to the schedule to this act any article or ingredient the addition of which is by him deemed necessary in the public interest; and every order in council in that behalf shall be published in the Canada Gazette, and shall take effect at the expiration of thirty days from the date of such publication. (48-49 V., c. 67 s. 18.)

19. The governor in council shall from time to time cause to be prepared and published lists of the articles, mixtures, or compounds declared exempt from the provisions of this act in accordance with the next preceding section, and shall also from time to time fix the limits of variability permissible in any article of food or drug or compound the standard of which is not established by any such pharmacopœia or standard work as is hereinbefore mentioned; and the orders in council fixing the same shall be published in the Canada Gazette, and shall take effect at the expiration of thirty days after the publication thereof. (48-49 V., c. 67, s. 19.)

20. Whenever any article of food, any drug, or any agricultural fertilizer is reported by any analyst as being adulterated within the meaning of this act, the minister of inland revenue may, if he thinks fit, order such article, and all other articles of the same kind and quality which were in the same place at the time the article analyzed was obtained, to be seized by any officer of customs or inland revenue, and detained by him until an analysis of samples of the whole is made by the chief analyst. (48, 49 V., c. 67, s. 20.)

21. If the chief analyst reports to the minister of inland revenue that the whole or any part of such articles are adulterated, the minister may declare such articles, or so much thereof as the chief analyst reports as being adulterated, to be forfeited to the Crown; and such articles shall thereupon be disposed of as the minister directs. (48-49 V., c. 67, s. 21.)

PENALTIES.

22. Every person who willfully adulterates any article of food or any drug, or orders any other person so to do, shall—

(a) If such adulteration is within the meaning of this act deemed to be injurious to health, for the first offense incur a penalty not exceeding fifty dollars and not less than ten dollars and costs, and for each subsequent offense a penalty not exceeding two hundred dollars and not less than fifty dollars and costs.

(b) If such adulteration is within the meaning of this act deemed not to be injurious to health, incur a penalty not exceeding thirty dollars and costs, and for each subsequent offense a penalty not exceeding one hundred dollars and not less than fifty dollars and costs. (49-49 V., c. 67, s. 22.)

23. Every person who, by himself or his agent, sells, offers for sale, or exposes for sale any article of food or any drug which is adulterated within the meaning of this act shall—

(a) If such adulteration is within the meaning of this act deemed to be injurious to health, for a first offense incur a penalty not exceeding fifty dollars and costs, and for each subsequent offense a penalty not exceeding two hundred dollars and not less than fifty dollars and costs.

(b) If such adulteration is within the meaning of this act deemed not to be injurious to health, incur for each such offense a penalty not exceeding fifty dollars and not less than five dollars and costs.

(2) *Provided*, That if the person accused proves to the court before which the case is tried that he did not know of the article being adulterated, and shows that he could not, with reasonable diligence, have obtained that knowledge, he shall be sub-

ject only to the liability to forfeiture under the twenty-first section of this act. (48-49 V., c. 67, s. 23.)

24. Every compounder or dealer in and every manufacturer of intoxicating liquors who has in his possession or in any part of the premises occupied by him as such any adulterated liquor, knowing it to be adulterated, or any deleterious ingredient specified in the schedule hereto, or added to such schedule by the governor in council, for the possession of which he is unable to account to the satisfaction of the court before which the case is tried, shall be deemed knowingly to have exposed for sale adulterated food, and shall incur for the first offense a penalty not exceeding one hundred dollars, and for each subsequent offense a penalty not exceeding four hundred dollars. (48-49 V., c. 67, s. 24.)

25. Every person who knowingly attaches to any article of food or any drug any label which falsely describes the article sold or offered or exposed for sale, shall incur a penalty not exceeding one hundred dollars and not less than twenty dollars and costs. (48-49 V., c. 67, s. 25.)

26. Every penalty imposed and recovered under this act shall, except as herein otherwise provided, and except in the case of any suit, action, or prosecution brought or instituted under the provisions of the next following section, be paid over to the minister of finance and receiver-general, and shall form part of the consolidated revenue fund. (48-49 V., c. 67, s. 26.)

GENERAL PROVISIONS.

27. Nothing herein contained shall be held to preclude any person from submitting any sample of food, drug, or agricultural fertilizer for analysis to any public analyst, or from prosecuting the vendor thereof, if such article is found to be adulterated, but the burden of proof of sale, and of the fact that the sample was not tampered with after purchase, shall be upon the person so submitting the same:

(2) Any public analyst shall analyze such sample on payment of the fee prescribed in respect of such article or class of article by the governor in council. (48-49 V., c. 67, s. 27.)

28. Any expenses incurred in analyzing any food, drug, or agricultural fertilizer, in pursuance of this act, shall, if the person from whom the sample is taken is convicted of having in his possession, selling, offering or exposing for sale, adulterated food, drugs, or agricultural fertilizers, in violation of this act, be deemed to be a portion of the cost of the proceedings against him, and shall be paid by him accordingly; and in all other cases such expenses shall be paid as part of the expenses of the officer, or by the person who procured the sample, as the case may be. (48-49 V., c. 67, s. 28.)

29. The governor in council may, from time to time, make such regulations as to him seem necessary, for carrying the provisions of this act into effect. (48-49 V., c. 67, s. 20.)

30. The provisions of "the inland revenue act," whether enacted with special reference to any particular business or trade, or with general reference to the collection of the revenue, or the prevention, detection, or punishment of fraud or neglect in relation thereto, shall extend, apply, and be construed, and shall have effect with reference to this act, as if they had been enacted with special reference to the matters and things herein provided for.

(2) Every penalty imposed under this act may be enforced and dealt with as if imposed under the said act, and every compounder, and the apparatus used by him, and the place in which his business is carried on, and the articles made or compounded by him, or used in compounding any such article, shall be "subject to excise" under the said act. (48-49 V., c. 67, s. 30.)

SCHEDULE.

Cocculus indicus, chloride of sodium (otherwise common salt), copperas, opium, cayenne pepper, picric acid, Indian hemp, strychnine, tobacco, daniel seed, extract of logwood, salts of zinc, copper, or lead, alum, methyl alcohol and its derivatives, amyl alcohol, and any extract or compound of any of the above ingredients.

The English laws will be found in Hassall or Blyth.

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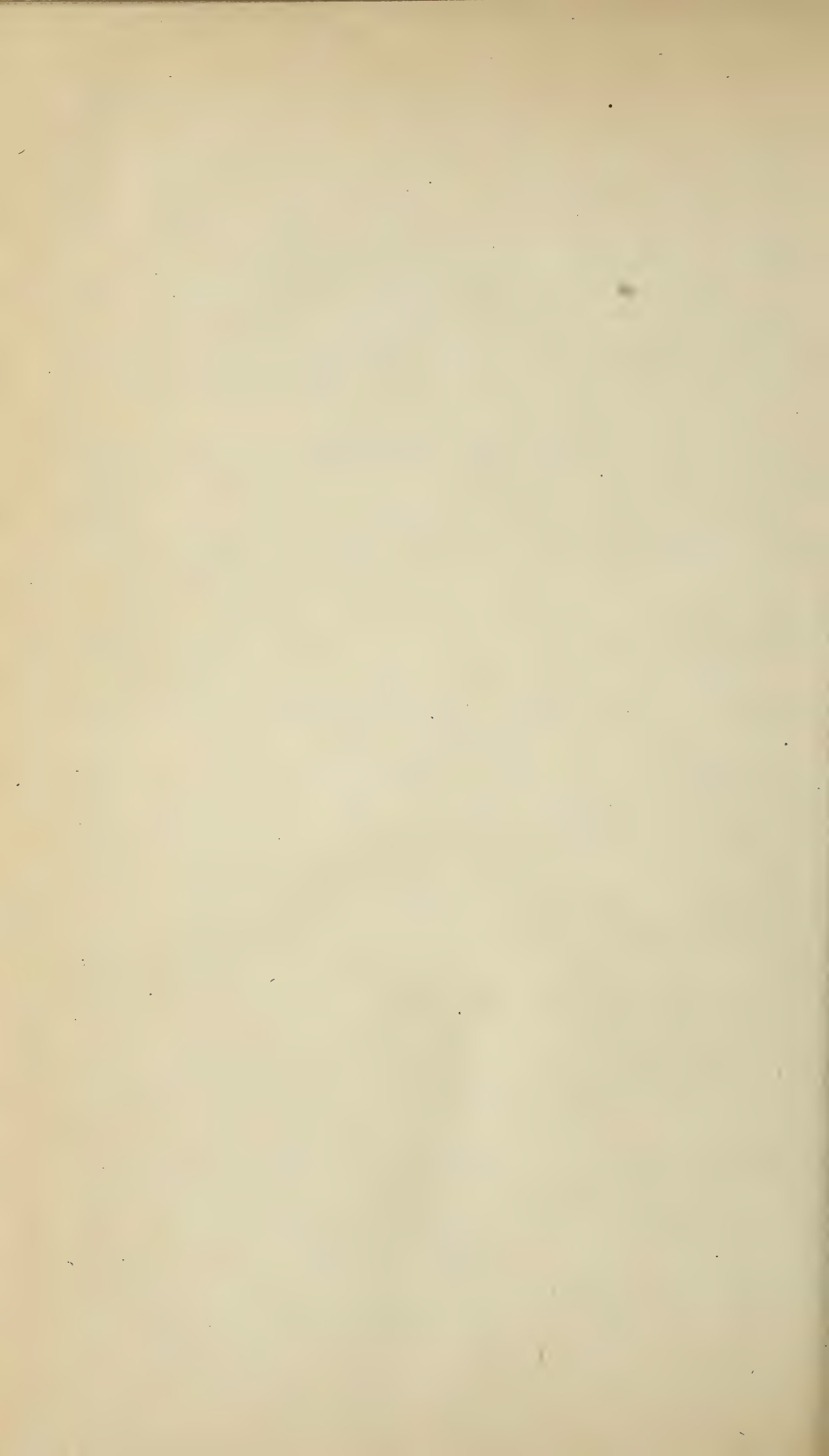
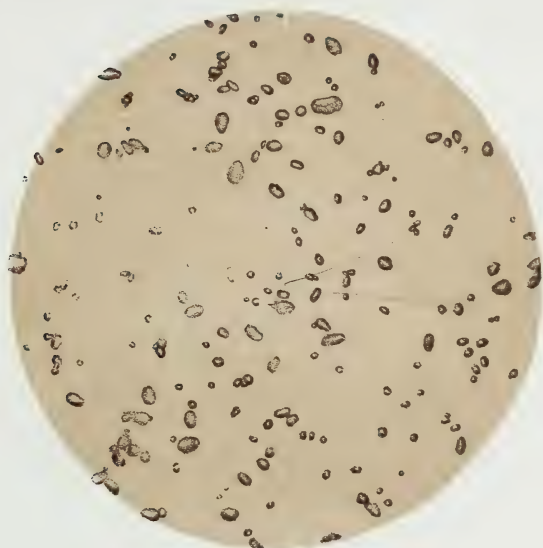


Fig 25



STARCH PLAIN ILLUMINATION x43

Fig 26



STARCH STAINED WITH IODINE x43

Fig 27



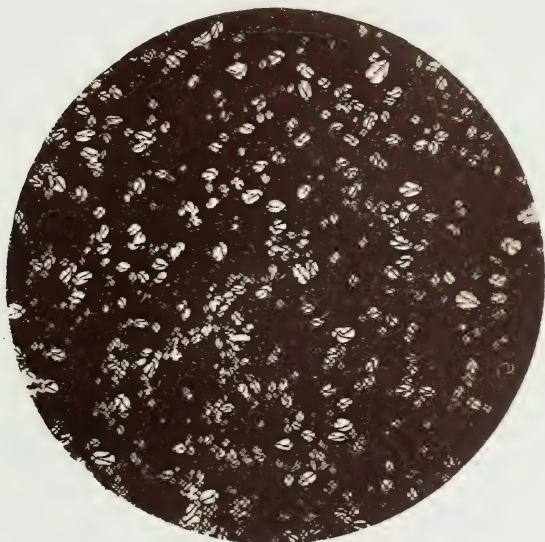
MARUNTA STARCH

Fig 28



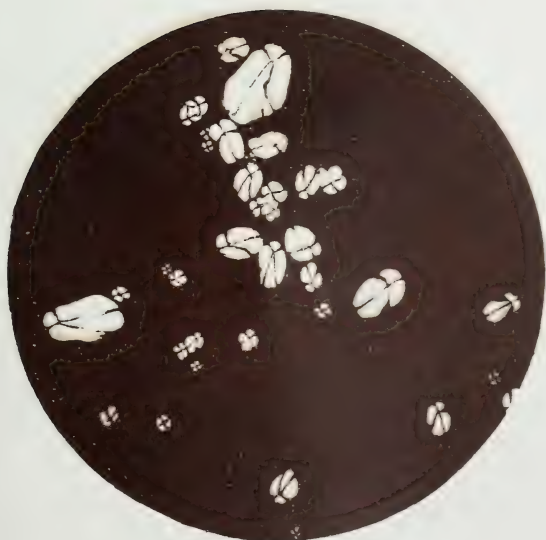
MARUNTA STARCH x140

Fig 29



POTATO STARCH x43

Fig 30



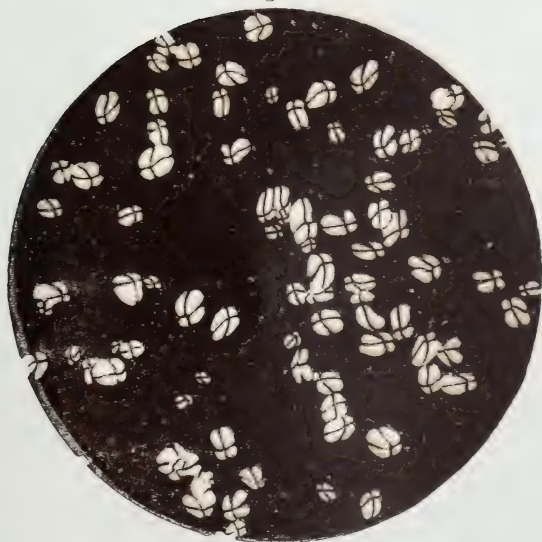
POTATO STARCH

Fig 31



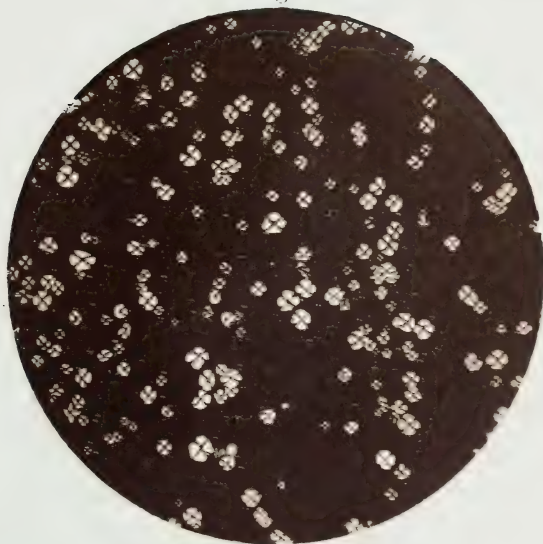
POTATO STARCH x144

Fig 32



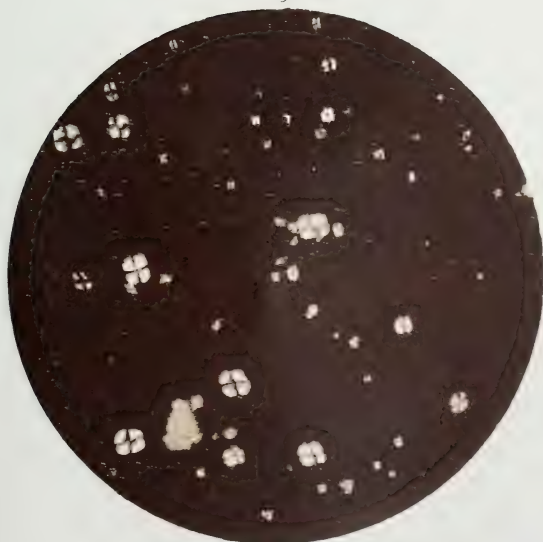
MARUNTA STARCH x144

Fig 33



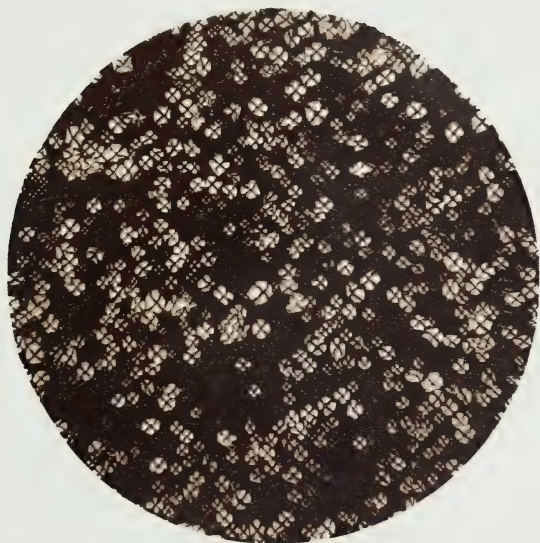
MAIZE STARCH $\times 145$

Fig 34



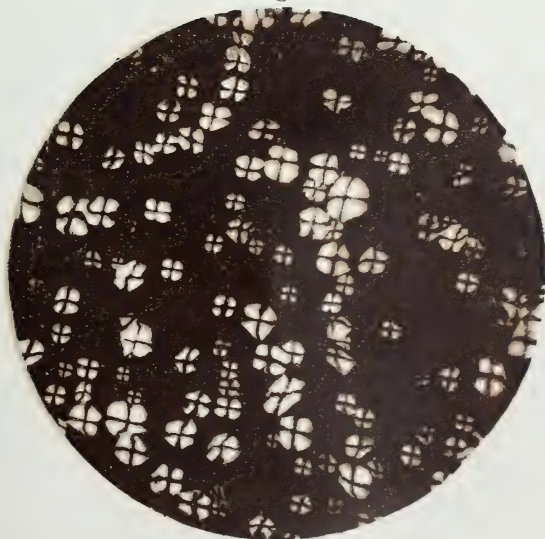
WHEAT STARCH $\times 145$

Fig 35



RICE STARCH x150

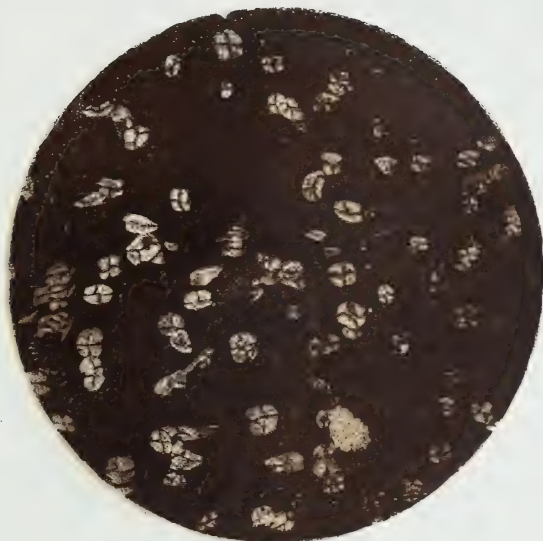
Fig 36



RICE STARCH x450



Fig 37



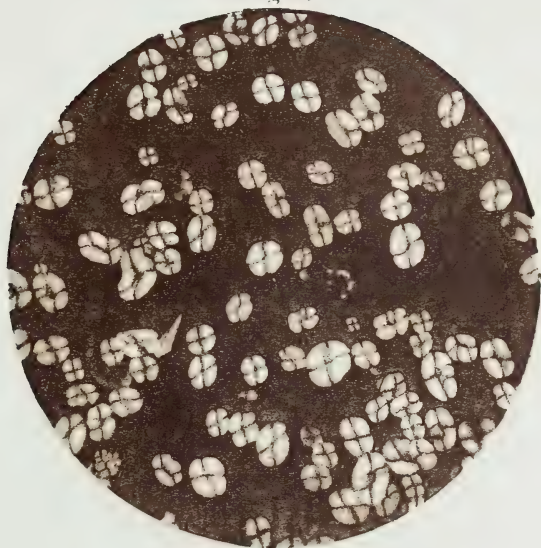
BARLEY STARCH x150

Fig 38



OAT STARCH x160

Fig 39



BEAN STARCH x145

Fig 40



PEA STARCH x145

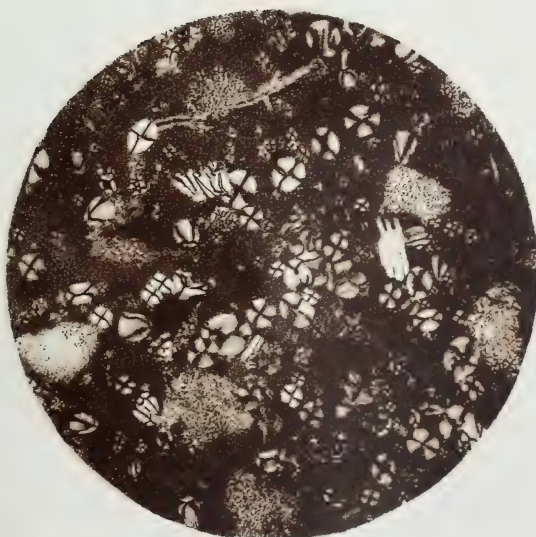


Fig 41



GINGER STARCH $\times 145$

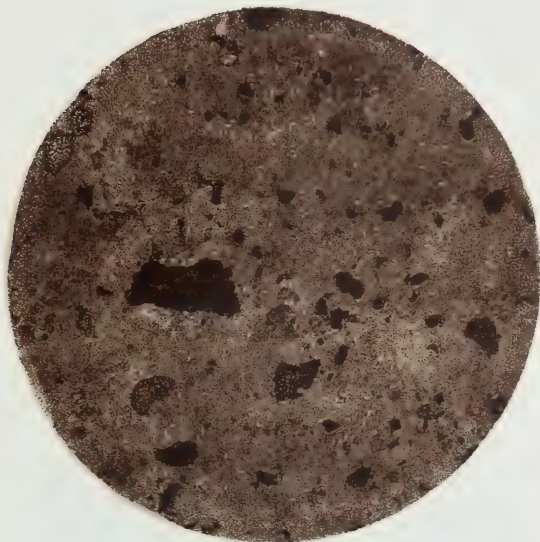
Fig 42



GINGER ADULTERATED $\times 145$

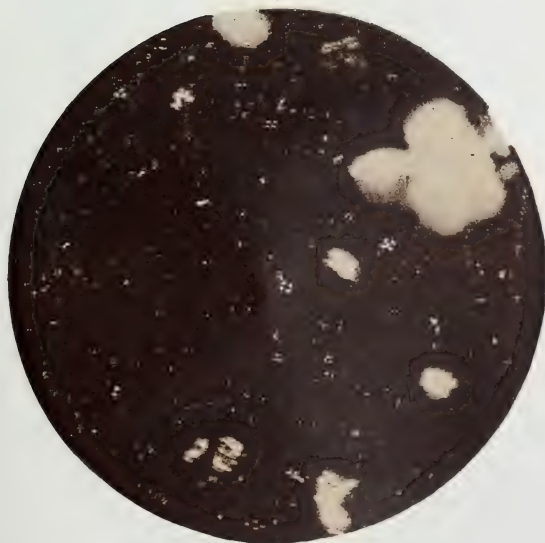


Fig 43



BLACK PEPPER P.D. $\times 45$

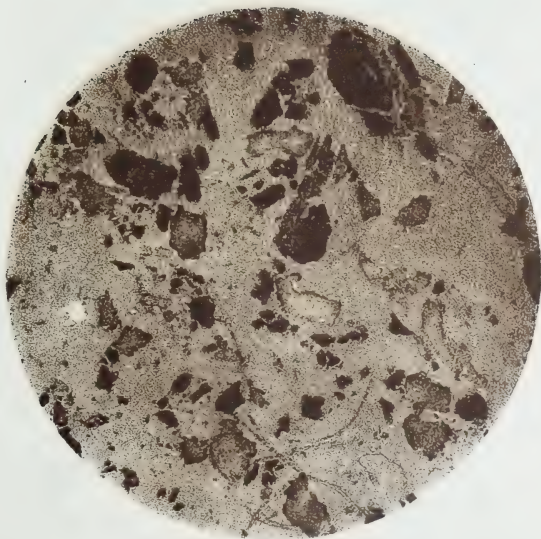
Fig 44



PEPPER ADULTERATED $\times 45$



Fig 45



CINNAMON ADULTERATED

Fig 46



CINNAMON x45

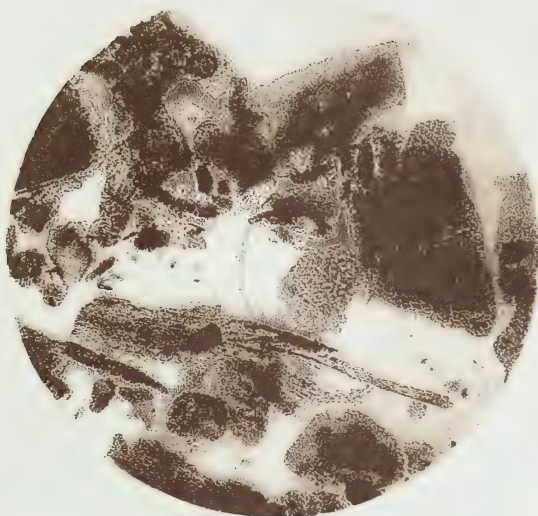
Fig 47



CASSIA x45



Fig 48

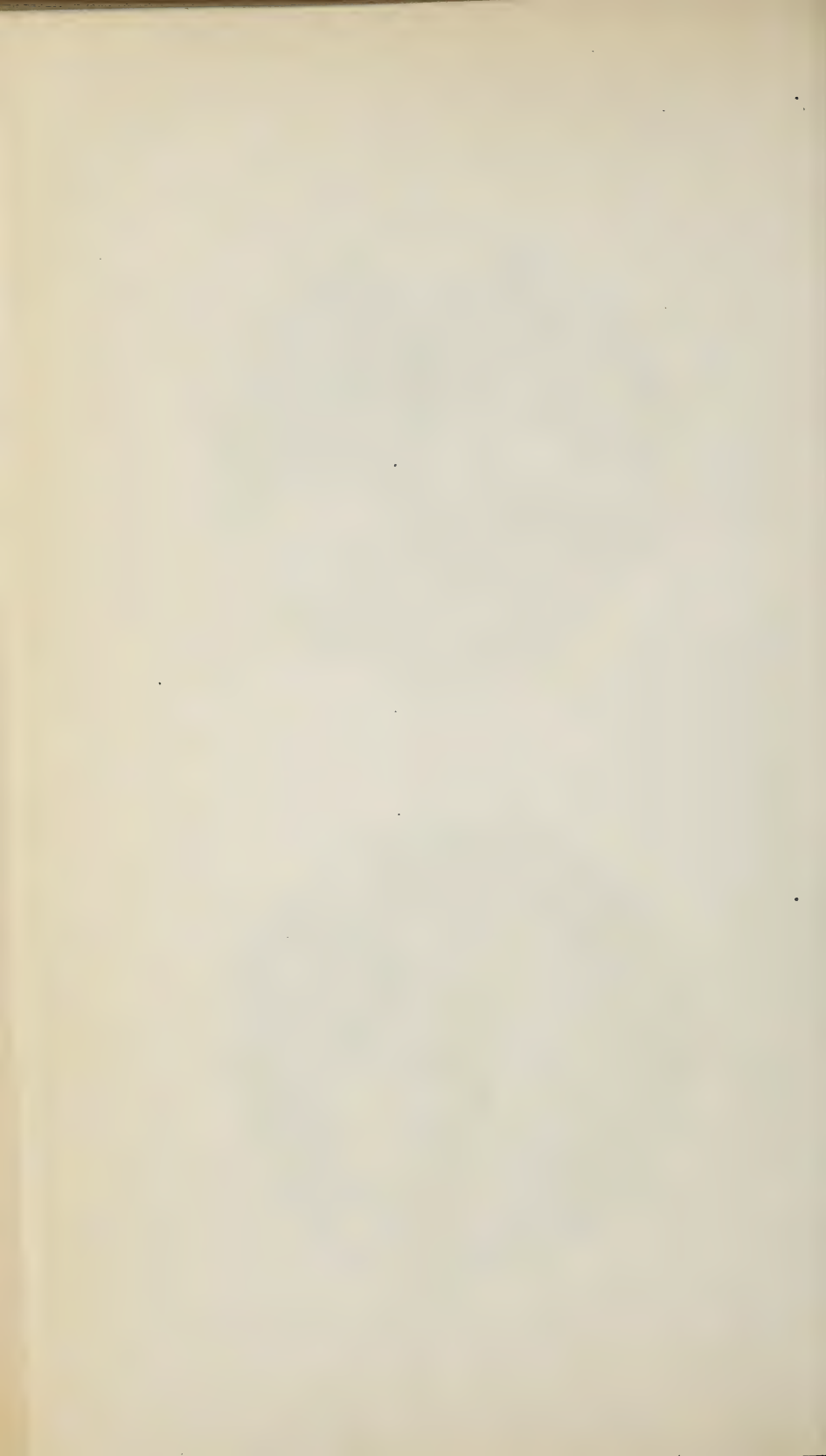


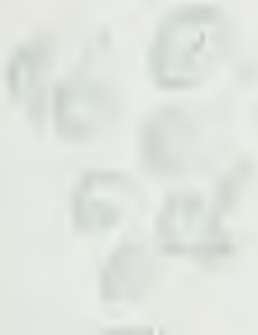
CAYENNE

Fig 49



CAYENNE ADULTERATED





EARLEY



IG. 55

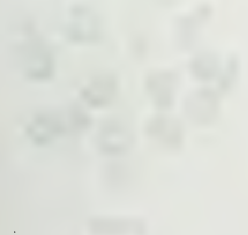






FIGURE 1.



FIGURE 2.



FIGURE 3.



FIGURE 4.

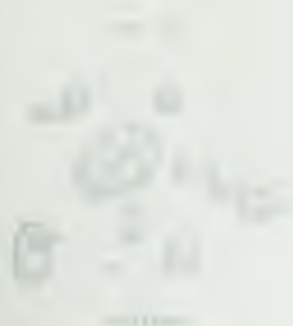




Micrograph 1



Micrograph 2



Micrograph 3



Micrograph 4



Micrograph 5



Micrograph 6



U. S. DEPARTMENT OF AGRICULTURE.

DIVISION OF CHEMISTRY.

BULLETIN

No. 13.

FOODS

AND

FOOD ADULTERANTS.

BY DIRECTION OF

THE COMMISSIONER OF AGRICULTURE.

PART THIRD:

FERMENTED ALCOHOLIC BEVERAGES,

MALT LIQUORS, WINE, AND CIDER.

BY

C. A. CRAMPTON,

ASSISTANT CHEMIST.

WASHINGTON:

GOVERNMENT PRINTING OFFICE.

1887.



[BULLETIN No. 13.]

PART 3.—FERMENTED ALCOHOLIC BEVERAGES.

LETTER OF TRANSMITTAL.

U. S. DEPARTMENT OF AGRICULTURE,

Washington, D. C., August 15, 1887.

SIR: I have the honor to submit herewith that portion of the work on food adulteration which has been under my charge.

I take this opportunity for acknowledging my indebtedness to the following gentlemen, Messrs. Trescot, Fake, Leugfeld, and Dugan—my fellow assistants in the division—by whom a large part of the analytical work was performed.

Respectfully,

C. A. CRAMPTON,

Assistant Chemist.

Dr. H. W. WILEY,

Chief Chemist.

4450—No. 13, PT. 3—1



LIST OF PRINCIPAL PUBLICATIONS CONSULTED.

The following is a list of the principal publications (with the exception of periodicals) that have been consulted in the course of this investigation :

- KOENIG.** Die menschlichen Nahrungs- und Genussmittel. Berlin, 1880.
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ELSNER. Die Praxis des Nahrungsmittel-Chemikers. Hamburg und Leipzig, 1882.
HILGER. Vereinbarungen betreffs der Untersuchung und Beurteilung von Nahrungs- und Genussmitteln. Herausgegeben im Auftrage der Freien Vereinigung Bayrischer Vertreter der angewandten Chemie. Berlin, 1885.
BARTH. Die Weinanalyse. Kommentar der im kaiserlichen Gesundheitsamte 1884 zusammengestellten Beschlüsse der Kommission zur Beratung einheitlicher Methoden für die Analyse des Weines. Hamburg und Leipzig, 1884.
MEYER und FINKELBURG. Das Gesetz betreffend den Verkehr mit Nahrungsmitteln, Genussmitteln und Gebrauchsgegenständen, vom 12. Mai 1879. Berlin, 1885.
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 Documents sur les falsifications des matières alimentaires et sur les travaux du Laboratoire Municipal, deuxième rapport. Paris, 1885. G. Masson, éditeur.
BLYTH. Foods: their composition and analysis. London, 1882.
HASSALL. Food: its adulterations and the methods for their detection. London, 1876.
ALLEN. Commercial organic analysis. 1 vol. 2d edition. London, 1886.
PRESCOTT. Chemical analysis of alcoholic liquors. New York.
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 Annual report of the Department of Health of the City of Brooklyn for 1885 and 1886. Brooklyn.
 University of California, College of Agriculture. Report of professor in charge, 1879, 1880, 1882, and 1884.—Report of viticultural work during the seasons 1883-'84 and 1884-'85, same 1885 and 1886. Sacramento.



PART I.

MALT LIQUORS.



MALT LIQUORS.

The production of malt liquors in this country as an industry is second only in importance to the production of breadstuffs. Their consumption is steadily on the increase, as is also the amount consumed in proportion to other kinds of alcoholic beverages. The following tables are taken from recent statistics, compiled by the Bureau of Statistics, U. S. Treasury Department, from figures obtained from official sources:¹

Annual consumption of distilled and malt liquors and wines in the United States and the average annual consumption per capita of population during the years 1840, 1850, 1860, and from 1870 to 1886, inclusive.

Year ending June 30—	Distilled spirits consumed.				Wines consumed.		
	Spirits of domestic product.		Imported spirits entered for consumption.	Total.	Wines of domestic product. ²	Imported wines entered for consumption.	Total.
	From fruit.	All other.					
	<i>Pr. galls.</i>	<i>Pr. galls.</i>	<i>Pr. galls.</i>	<i>Pr. galls.</i>	<i>Gallons.</i>	<i>Gallons.</i>	<i>Gallons.</i>
1840	(³) 40,378,090		2,682,794	43,060,884	124,734	4,748,362	4,873,096
1850	(³) 46,768,083		5,065,390	51,833,473	221,249	6,094,622	6,315,871
1860	(³) 83,904,258		6,064,393	89,968,651	1,860,008	9,199,133	11,059,141
1870	1,223,820	77,266,368	1,405,510	79,895,708	3,059,518	9,165,549	12,225,067
1871	2,472,011	59,842,617	1,745,033	64,059,661	4,980,783	10,853,280	15,834,063
1872	1,089,698	65,145,880	2,186,702	68,422,280	6,968,737	9,713,300	16,682,037
1873	2,965,987	62,945,154	2,125,998	68,037,129	8,953,285	9,893,746	18,847,031
1874	766,687	61,814,875	1,958,528	64,540,090	10,951,809	9,516,855	20,468,714
1875	1,757,202	62,668,709	1,694,647	66,120,558	12,954,961	7,036,369	19,991,330
1876	672,221	57,340,472	1,471,197	59,483,890	14,968,085	5,193,723	20,161,808
1877	1,527,141	57,016,248	1,376,729	59,920,118	16,942,592	4,933,738	21,876,330
1878	1,103,351	49,600,838	1,227,752	51,931,941	17,953,386	4,310,563	22,263,949
1879	1,021,708	52,003,467	1,253,300	54,278,475	19,845,113	4,532,017	24,377,130
1880	1,005,781	61,126,634	1,394,279	63,526,694	23,298,940	5,030,601	28,329,541
1881	1,701,206	67,426,000	1,479,875	70,607,081	18,931,819	5,231,106	24,162,925
1882	1,216,850	70,759,548	1,580,578	73,556,976	19,934,856	5,628,071	25,562,927
1883	1,253,278	75,508,785	1,690,624	78,452,687	17,406,028	8,372,152	25,778,180
1884	1,137,056	78,479,845	1,511,680	81,128,581	17,402,938	3,105,407	20,508,345
1885	1,468,775	67,689,250	1,442,067	70,600,092	17,404,698	4,495,759	21,900,457
1886	1,555,994	69,295,361	1,410,259	72,261,614	17,366,393	4,700,827	22,067,220

¹ Statements Nos. 32 to 50, inclusive, of the Quarterly Report No. 2 series 1886-'87, of the Chief of the Bureau of Statistics, Government Printing Office, 1887.

² Product less exports.

³ Included with "All other."

Annual consumption of distilled and malt liquors and wines, &c.—Continued.

Year ending June 30—	Malt liquors consumed.			Total consumption of wines and liquors.	Total consumption per capita of population.			
	Malt liquors of domestic product. ¹	Imported malt liquors entered for con- sumption.	Total.		Dis- tilled spirits.	Wines.	Malt liquors.	All wines and liquors.
	Gallons.	Gallons.	Gallons.	Gallons.	Pr. galls.	Gallons.	Gallons.	Gallons.
1840	23, 162, 571	148, 272	23, 310, 843	71, 244, 817	2.52	0.29	1.86	4.17
1850	36, 361, 708	201, 301	36, 563, 009	94, 712, 353	2.23	0.27	1.58	4.08
1860	100, 225, 879	1, 120, 790	101, 346, 699	202, 374, 461	2.86	0.35	3.22	6.43
1870	203, 743, 401	1, 012, 755	204, 756, 156	296, 876, 931	2.07	0.32	5.30	7.69
1871	239, 838, 137	1, 299, 990	241, 138, 127	321, 031, 851	1.62	0.40	6.09	8.11
1872	268, 357, 983	1, 940, 933	270, 298, 916	355, 403, 233	1.68	0.41	6.65	8.74
1873	298, 519, 675	2, 177, 587	300, 697, 262	387, 581, 432	1.63	0.45	7.27	9.29
1874	297, 519, 981	2, 001, 084	299, 521, 065	384, 529, 869	1.51	0.48	6.99	8.98
1875	292, 961, 047	1, 992, 110	294, 953, 157	381, 065, 045	1.50	0.45	6.71	8.66
1876	306, 852, 467	1, 483, 920	308, 336, 387	387, 982, 085	1.32	0.45	6.83	8.60
1877	303, 854, 988	1, 072, 679	304, 926, 667	386, 723, 115	1.29	0.47	6.58	8.34
1878	317, 136, 597	832, 755	317, 969, 352	392, 165, 242	1.09	0.47	6.68	8.24
1879	343, 724, 971	880, 514	344, 605, 485	423, 261, 090	1.11	0.50	7.05	8.66
1880	413, 208, 885	1, 011, 280	414, 220, 165	506, 076, 400	1.26	0.56	8.26	10.08
1881	442, 947, 664	1, 164, 505	444, 112, 169	538, 882, 175	1.37	0.47	8.63	10.47
1882	524, 843, 379	1, 536, 501	526, 379, 980	625, 499, 883	1.39	0.48	9.97	11.81
1883	549, 616, 338	1, 881, 002	551, 497, 340	655, 728, 207	1.45	0.48	10.18	12.11
1884	588, 005, 609	2, 010, 908	590, 016, 517	691, 653, 443	1.46	0.37	10.62	12.45
1885	594, 063, 095	2, 068, 771	596, 131, 866	688, 632, 415	1.24	0.38	10.44	12.06
1886	640, 746, 288	2, 221, 432	642, 967, 720	737, 296, 554	1.24	0.38	11.18	12.62

¹ Product less exports.

NOTES.—(1) The data as to product of domestic liquors and wines for 1840, 1850, and 1860 were derived from the Census. (2) The consumption of imported liquors and wines for 1840, 1850, and 1860 is represented by the net imports. (3) The production of domestic wines, from 1870 to 1885, has been estimated by the Department of Agriculture: by Mr. Charles McK. Leoser, president of Wine and Spirit Traders' Society, New York, and other well-informed persons, and the amount stated as consumed represents the production minus the exports. (4) The consumption of domestic spirituous and malt liquors, from 1870 to 1886, was obtained from the reports of the Commissioner of Internal Revenue. (5) In computing the quantity of sparkling and still wines and vermouth in bottles, 5 so-called quart bottles are reckoned as equivalent to the gallon. (6) The consumption of distilled spirits as a beverage is estimated to be about 90 per cent. of the product consumed for all purposes.

This table shows admirably the rapid increase, especially in the last ten years, of the consumption of malt liquors, and the relative decrease in the consumption of the stronger alcoholic beverages. Thus it will be seen that in 1840 the amount of malt liquor consumed per capita was a little over one-half the amount of distilled liquor consumed; while in 1886 it was *nine times as much*. The amount of distilled liquor consumed per capita has diminished during the twenty-six years to one-half, while the amount of malt liquor consumed has increased very nearly seven times; or, in other words, the malt liquors have been driving out the distilled at the rate of about .05 gallons per capita each year, and supplanting it at the rate of about .38 gallons per capita.

The average quantity consumed annually for the last three years was 609,705,367 gallons, of which 2,100,370 gallons were imported. Taking this as a basis, Mr. F. N. Barrett, in the publication above mentioned, estimates the amount expended for beer per annum at \$304,852,683, placing the cost to the consumer at 50 cents per gallon. The cost to the consumer of the total quantity of liquors per annum he places at \$700,000,000.

That there is still opportunity for increase in the consumption of malt liquors in the United States will be seen from the following compara-

tive tables, from which it appears that while the United States quite holds her own in the quantity of distilled liquors consumed, she is still far behind the other great nations in the consumption of the milder alcoholic liquors.

Comparative summary of the consumption per capita of population in the United States, the United Kingdom, France, and Germany, of distilled spirits, wines, and malt liquors during each year from 1881 to 1885, inclusive.

[From original official data.]

Years.	Distilled spirits.				Wines.				Malt liquors.			
	United States.	United King. dom.	France.	Germany.	United States.	United King. dom.	France.	Germany.	United States.	United King. dom.	France.	Germany.
	Galls.	Galls.	Galls.	Galls.	Galls.	Galls.	Galls.	Galls.	Galls.	Galls.	Galls.	Galls.
1881.....	1.39	1.00	.94	1.32	.48	.43	18.52	(¹)	9.97	33.90	(¹)	22.35
1882.....	1.45	1.07	1.08	1.14	.48	.41	27.10	(¹)	10.18	33.65	(¹)	22.45
1883.....	1.46	1.03	1.24	1.19	.37	.40	30.75	(¹)	10.62	33.13	(¹)	22.45
1884.....	1.24	1.05	1.25	1.11	.38	.39	30.67	(¹)	10.44	32.72	(¹)	23.19
1885.....	1.24	1.01	1.32	1.14	.38	.37	36.88	(¹)	11.18	32.79	(¹)	23.78

¹No data.

NOTE.—The years referred to are, for France and Great Britain, calendar years; for the United States, the five years ending June 30, 1886; for Germany in the case of beer, the five years ending March 31, 1885, and in the case of spirits the five years ending March 31, 1881, these being the latest years for which data were obtainable.

It is hardly necessary, after the above showing, to dwell upon the importance of this article of daily consumption, or the necessity of a thorough acquaintance with its manufacture, composition, and the nature and extent of its adulterations. There is no beverage that compares with it in the amount consumed by the people except water, and possibly milk. But little supervision has been exercised over its manufacture and sale, except the rigorous enforcement by the Government of its demands for a share in the profits of its manufacture.

THE PROCESS OF BREWING.

Brewing, or the art of preparing an alcoholic drink from starchy grains by fermentation, is of very ancient origin. It was practiced by the Egyptians, and the Greeks and Romans learned the art from them. Herodotus speaks of the Egyptians making wine from corn, and it was undoubtedly practiced by the Greeks in the fifth century before Christ, as the use of malt beverages is mentioned in the writings of Æschylus and Sophocles, poets of that period. It is also mentioned by Xenophon, 400 B. C. The Romans are also supposed to have derived a knowledge of the art from the Egyptians, and Pliny and Tacitus both speak of its use among the Gauls and Germans of Spain and France.

It is supposed that the art was introduced into Britain by the Romans and acquired from the natives by the Saxons. According to Verstigan "this excellent and healthsome liquor, beere, anciently called ale,

as of the Danes it yet is, was of the Germans invented and brought into use." Ale-houses are mentioned in the laws of Ina, king of Wessex, A. D. 680. Ale-booths were regulated by law A. D. 728.

The art of producing an alcoholic drink from starchy seeds seems to have been nearly as extensively known and practiced among the various nations of the earth as the less complex operation of preparing a fermented liquor from the juice of fruits and plants containing sugar. Thus the Kaffre races of South Africa are said to have prepared for many years a malt liquor from the seeds of the millet (*Sorghum vulgare*) going through all the processes of germinating the seed, extracting the malt, and fermenting the wort. In the north of Africa another seed is used. The Chinese prepared the drink called sam shee from rice.

The process of brewing consists of two distinct operations: the malting and the brewing proper. In fact the two operations are frequently separated, many small breweries buying their malt ready prepared. When kept dry it retains its qualities for an indefinite period and is handled as an article of commerce.

MALTING.

The object of this operation is the germination of the grain, and the consequent formation of the ferment diastase, which shall subsequently, under the proper conditions, perform its specific function of converting the starchy portions of the grain into saccharine or fermentable matter. Barley is the grain used almost exclusively for this purpose, its advantages having been recognized even by the Egyptians; they seem to be principally of a physical character, consisting of the firmness of the kernel, and the hard husk, which freely allows the entrance of water, but prevents the passage of starch or insoluble matter.

The operations through which the grain is successively passed are called, technically, steeping, crushing, flooring, and kiln-drying. In the first it is spread out in large vats, covered with water, and allowed to steep several days. When it has become softened, the water is run off and the swollen grain is subjected to a slight degree of heat, which causes it to germinate. This is the second operation. The operation of flooring has for its end the regulation of the germination of the grain, and the time when it has progressed sufficiently is judged by the length which has been attained by the acrospire or plumule. This is variously given as from two-thirds to seven-eighths the length of the grain. The sprouted grain is now spread out in the malt kilns and heat applied, while a current of air circulates about it. After the moisture is driven off, which is done at a low temperature, about 90° F., the heat is raised, and finished at from 125° to 180° F., according to the grade of malt required, the difference between pale, amber, and brown malt being due simply to the temperature at which they are kiln-dried. This last operation serves not only to drive off the moisture, but also stops ger-

mination by destroying the vitality of the germ, and fits it for keeping. It also probably develops the flavor by the formation of a minute quantity of empyreumatic oil in the husk.

The rootlets and germs are removed in this process by the turning and stirring of the grain. The water which is used in the process of steeping the grain is an important factor in the production of good malt, and the preference of brewers for hard lime waters for this purpose has been shown by recent experiment to be rational, for it is found that when barley is steeped with distilled water, a very putrescible liquor is obtained charged with albuminous matter, while if a hard water is used these matters remain in an insoluble condition in the grain.

Chemically considered a good malt should not contain more than 5 per cent. of water, and the soluble extract should constitute about 70 per cent. of the weight of the malt and should have a good diastatic action on starch mucilage. The determination of the acidity is important in determining the keeping qualities of the beer which is to be brewed from it, and should not be over .3 per cent. (calculated as lactic).

The following analyses by O'Sullivan show the composition of pale malt:

	(1)	(2)
Starch	44.15	45.13
Other carbohydrates (of which 60-70 per cent. consist of fermentable sugars). Inulin (?) and a small quantity of other bodies soluble in cold water.....	21.23	19.39
Cellulose matter.....	11.57	10.09
Fat	1.65	1.96
Albuminoids.....	13.09	13.80
Ash	2.60	1.92
Water	5.83	7.47
	100.12	99.76

BREWING.

Brewing proper includes a number of distinct operations, such as grinding and mashing the malt, boiling and cooling the wort or infusion, fermenting it, and clearing and racking the beer. In the process of mashing takes place the conversion of the starch into fermentable sugar, mainly maltose, by the action of the diastase. Two methods are used for extracting the soluble matter from the malt, called *infusion* and *decoction*, respectively; the former is the method most in use in England, the latter in Germany and France. The wort prepared by infusion contains less dextrin and more albuminoid matter than that prepared by decoction; the beers from the former are stronger in alcohol, but not so good in keeping qualities.

A good wort should give no blue color with iodine, showing the complete conversion of all the starch, and should contain a large percentage of maltose, which should constitute about 70 per cent. of the extract.

After the mashing process comes the *boiling* of the wort, which is begun as soon as it is drawn off from the exhausted malt and continued for one to two hours. This prevents the formation of acid, and serves to extract the hops, which are added at this stage of the process. The boiling of the wort with hops serves not only to impart to it the desired hop flavor, but also to partially clarify it by precipitating some albuminous matter by means of the tannin in the hops, and to enhance its keeping qualities. To this end larger quantities of hops are used for beers intended for exportation or long keeping.

The wort is now ready to be submitted to the most important operation of all—fermentation—which calls for very careful supervision on the part of the brewer.

FERMENTATION.

After the wort has been boiled with hops it is cooled as rapidly as possible, to prevent the formation of acid, usually effected by means of artificial refrigerating apparatus; it is then ready for the addition of the yeast.

There are two distinct methods of fermentation in use, called by the Germans *Ober- und Untergährung*, and by the French *fermentation haute* (top fermentation) and *basse* (bottom fermentation). The former is carried on at a comparatively high temperature, the action is rapid, and the yeast with the impurities is carried to the surface of the liquid; in the latter method the temperature is kept low, the fermentation goes on slowly, and the yeast and impurities sink to the bottom. The second method is often called the Bavarian method, as it seems to have originated there, and is used exclusively in that country. It is generally preferred in Germany and France, while in England and this country the upward clearing method appears to be more in vogue.

The nature of the fermentation depends greatly upon the character of the yeast used, for Pasteur's experiments have shown that yeast from upward-fermented beer tends to produce the upward fermentation, while yeast from bottom-fermented beer produces the bottom fermentation. The purity of the yeast used is of the very first importance in the production of good beer. Many experiments have been made with the end in view of producing a perfectly pure yeast, which should contain only the yeast ferment proper, and thus produce a beer of good flavor and keeping properties, free from diseased or acid ferments.

PURE YEAST.

The production of pure yeast for brewing purposes has been put on a practical basis of late years through the scientific researches of Dr. E. C. Hansen, of the Carlsberg Institute, in Copenhagen. He succeeded in producing a pure yeast cultivated from a *single cell*. He was able to differentiate in this way six different species or varieties of *saccharomyces*, several of which may usually be found in an ordinary brewery

yeast. These different varieties have been shown to produce beers differing not only in coloring, flavoring, facility of separation of the yeast, &c., but also in chemical composition.

In a recent address before the Society of Chemical Industry by G. H. Morris,¹ a résumé is given of the work done in this direction by Hansen and others.

Dr. Morris states that the employment of the pure yeasts is coming very largely into use in the beer-drinking countries of the Continent, and has met with favor from some of the most noted brewing technologists, such as Jacobson, Aubry, März, and Lintner, the latter of whom sums up the question in the following statements:

- (1) By contamination with so-called wild yeast an otherwise normal brewery yeast can be rendered incapable of producing a beer of good flavor and with good keeping qualities.
- (2) A contamination with wild yeasts may be produced by the dust of the air during summer and autumn, by the malt, or other sources.
- (3) By employing Hansen's method of pure cultivation and analysis it is possible to obtain from a contaminated yeast a good brewery yeast in a state of purity.
- (4) Yeast cultivated in a state of purity possesses in a marked degree the properties of the original yeast before contamination as far as concerns the degree of alteration of the flavor and keeping qualities of the beer.
- (5) There exist different varieties of normal bottom yeast (*S. cerevis.*), each with special properties which, like the peculiarities of species, are maintained constant.

The use of this yeast has not yet extended to England, although experiments on an industrial scale are now being carried on at Burton-on-Trent with different species of pure yeast.

The chemical characteristics of beer made from the different species of pure yeast have been investigated by Borgmann,² who analyzed samples of beer produced from two species of pure yeast, each cultivated from a single cell and the beer fermented under comparable conditions. The analysis gave the following results:

	Beer prepared with—	
	Yeast No. 1 gives in 100 cc.	Yeast No. 2 gives in 100 cc.
Alcohol.....	4.13	4.23
Extract.....	5.35	5.84
Ash.....	.20	.25
Free acid (as lactic).....	.086	.144
Glycerol.....	.109	.137
Phosphoric acid.....	.0775	.0828
Nitrogen.....	.071	.0719

From these numbers, which are the means of many determinations, the analyst concludes that the different yeasts produce beers which differ in chemical composition. He also finds that the proportion of alcohol

¹ Jour. Soc. Chem. Ind., 1887, p. 113.

² Zeit. Anal. Chem. 25, 532.

to glycerol is different from that formed with other beers. From analyses of other beers he finds that the proportion is—

	Alcohol.	Glycerol.
Maximum	100	5.497
Minimum	100	4.14

while with the Carlsberg pure yeast the proportion is—

	Alcohol.	Glycerol.
No. 1	100	2.63
No. 2	100	3.24

BELGIAN BEERS.

The method of brewing pursued in Belgium would seem the most unscientific known, still the Belgium beers are largely consumed in Europe. No yeast whatever is used, but the wort is left to ferment of itself, as it were, standing in the vats at a low temperature, until fermentation is provoked by germs that have accidentally fallen into it, or which may have found their way in during the process of manufacture. The action is naturally very slow, requiring sometimes several years for its completion, and a considerable formation of acid takes place, which is a predominant constituent in this class of beers.

CLARIFYING, STORING, AND PRESERVING.

The treatment of malt liquors after the process of fermentation is complete is very diverse, according to the kind of liquors it is intended to produce, the length of time it is to be kept, &c. The problem of clarifying and preserving the beer is very simple of solution if it has been properly and carefully brewed, for then it is easily cleared and keeps well; but where the reverse is the case it is necessary to make use of various clarifying and preserving agents, and here comes in the delicate question of the proper agents to use, which will perform this duty and still introduce no objectionable constituents into the drink.

The discussion of this question comes properly under the head of adulterations, and will be considered later on. As clarifying agents may be mentioned gelatine, tannin, Iceland moss, and flaxseed, and as mineral coagulating agents phosphate of lime, and alum.

Formerly beer was stored in casks or vats in cool cellars for a long period, to allow it to age or ripen, especially in Germany, whence came the name of "lager" beer, but the aim of the brewer at the present day is to produce an article fit for the market in as short a time as possible and thus turn his capital often and keep step with the rapid pace of modern business industry, so that the name of lager beer is rather a misnomer.

COMPOSITION OF MALT LIQUORS.

The composition of malt liquors varies greatly according to the materials used, the method of brewing, the season, and the use for which it is intended.

Malt liquors, properly so called, should be made only of malted barley, hops, yeast, and water, but the use of other materials as substitutes for the first three ingredients has extended so greatly in countries where their use is not prohibited that it is difficult to define what a beer really is.

Modern beer has been defined as a "fermented saccharine infusion to which some wholesome bitter has been added."

Its chemical composition is very complex, the principal constituents being alcohol, various sugars and carbohydrates, nitrogenous matter, carbonic, acetic, succinic, lactic, malic, and tannic acids, bitter and resinous extractive matter from the hops, glycerine, and various mineral constituents, consisting mainly of phosphates of the alkalies and alkali earths.

VARIETIES.

The names given to different kinds of malt liquors relate to various attributes, as the country where they were produced, as English, German, Bavarian beer, &c., or to the peculiarities in the method of brewing, &c. Thus, *porter* is simply a beer of high percentage of alcohol, and made from malt dried at a somewhat high temperature, which gives its dark color; *ale* is a pale beer, likewise of high attenuation and made of pale malt, with more hop extract than *porter*. *Stout* has less alcohol and more extract and still less hops than *porter*. These terms are used chiefly with reference to English malt liquors. The terms used for German beers, such as *Erlanger*, *Münchener*, &c., are for the most part names of places and are applied to beers made in imitation of the beers originally brewed in those cities. *Export* beer is beer that is specially prepared with a view to long-keeping qualities.

The following table gives some recently published and very complete analyses of beers made by C. Graham :¹

¹ Jour. Soc. Chem. Ind.

Composition of various typical beers (Graham).

	Non-volatile.						Volatile.*		Total nitrogen (by soda lime).	Specific gravity of beer.	Original gravity of wort.	Ratio of maltose to dex- trine.	Ratio of solids to alcohol.
	Maltose.	Dextrine.	Albuminoids (Wank- lynized).	Lactic and succinic acids.	Ash, coloring matter, and hop extract (by difference).	Total solid matters.	Acetic acid.	Alcohol.					
Burton ales:													
Mild	2.13	3.64	0.26	0.18	0.53	6.74	0.01	6.78	1080.0	1:1.7	1:1	
Pale	1.75	2.48	0.21	0.14	0.55	5.13	0.02	5.37	1062.0	1:1.4	1:1.05	
Bitter	1.62	2.60	0.16	0.17	0.87	5.42	0.01	5.44	1061.0	1:1.6	1:1	
Mild X	1.87	1.88	0.20	0.14	1.30	6.39	0.04	4.60	1055.5	1:1	1:0.85	
XXX	2.88	2.04	0.30	0.10	1.48	6.80	0.02	6.50	1073.7	1:0.7	1:0.96	
A. K. bitter	0.81	0.75	0.21	0.14	0.85	2.76	0.06	4.69	1044.6	1:1	1:1.7	
Somersetshire ale two years old	1.54	2.48	0.42	0.04	0.94	6.02	0.07	6.50	1071.0	1:1.6	1:1.08	
Scotch export, bitter	1.62	2.50	0.30	0.09	0.70	5.21	0.16	5.00	1057.0	1:1.5	1:0.96	
Dublin stout, XX	3.45	3.07	0.26	0.17	1.76	8.71	0.01	5.50	1074.0	1:0.88	1:0.63	
Dublin stout, XXX	5.35	2.09	0.43	0.25	1.40	9.52	0.04	6.78	1089.0	1:0.39	1:0.71	
German beer:													
Vienna lager	1.64	2.74	0.36	0.13	1.12	5.90	0.02	4.69	0.134	1016.5	1:1.66	1:0.78	
Pilsen lager	0.69	2.65	0.20	0.09	0.59	4.22	0.02	3.29	0.090	1011.8	1:3.8	1:0.8	
Munich lager	1.57	3.15	0.40	0.14	1.82	7.08	0.01	4.75	0.400	1021.0	1:2	1:0.67	
Tivoli (sold in London)	1.75	2.15	0.17	0.12	1.39	5.58	0.02	5.31	0.060	1014.0	1:1.23	1:0.95	
Bohemian (average)	0.57	2.14	3.97	3.81	1008.0	1:3.75	1:0.96	
Bavarian (average)	0.87	3.26	6.17	4.14	1016.0	1:3.75	1:0.67	

* Water not determined.

The following is taken from the report of the Municipal Laboratory Paris for 1885, and gives the composition of the principal beers sold in Paris.¹

Analyses of beers made in Municipal Laboratory of Paris in 1881.

No.	Density.	Per cent. by volume of alcohol.	Grams per liter.						Polarization.	Remarks.
			Extract.	Sugar.	Dextrin.	Albuminoid.	Ash.	Acidity as H ₂ SO ₄ .		
1.....	1.020	6.1	64.20	12.50	34.10	3.04	2.28	+15°.76	Good.
2.....	1.020	6.2	65.00	15.00	40.60	3.86	2.25	+16°.25	
3.....	1.019	6.2	63.10	13.17	28.20	+ 5°.88	
4.....	1.021	5.5	65.70	12.35	31.80	2.33	+ 7°.38	Contained salicylic acid. Good.
5.....	1.017	6.2	56.20	13.00	2.39	+14°.50	
6.....	1.022	5.0	75.90	11.62	47.16	2.32	+ 7°.50	
7.....	1.024	5.6	79.25	17.32	32.91	2.32	1.47	+17°.48	Contained salicylic acid. Good.
8.....	1.027	6.1	62.25	11.16	51.09	2.09	.68	+15°.34	
9.....	1.030	5.8	98.36	23.92	36.43	2.93	1.47	+23°.52	
10.....	1.019	5.2	65.53	12.56	2.04	1.12	+14°.20	Contained salicylic acid. Good.
11.....	1.020	5.7	65.50	15.83	1.58	1.96	+18°.05	
12.....	1.017	6.5	58.72	11.58	1.64	2.45	+15°.17	
13.....	1.026	4.0	58.42	10.01	1.94	2.45	+12°.21	Contained salicylic acid. Good.
14.....	1.020	5.7	63.40	8.30	+ 6°.27	
15.....	5.8	53.48	14.29	2.70	+14°.50	
16.....	1.013	5.9	59.59	9.81	2.90	1.21	+12°.41	Contained salicylic acid. Good.
17.....	1.014	5.9	60.05	9.79	2.95	1.12	+11°.54	
18.....	1.019	5.3	48.90	8.28	2.85	2.03	+ 7°.74	
19.....	7.3	78.45	16.54	4.30	+20°.00	Passable. Good.
20.....	4.7	58.71	12.23	2.90	1.04	+20°.68	
21.....	3.4	27.4595	1.36	+ 5°.60	
22.....	3.8	64.49	11.91	2.35	.86	+13°.12	

General averages of 135 samples of beer, analyzed in 1882: Alcohol, 4.25 per cent. by volume; extract, 52.06 grams per liter.

The following, taken from the same source, is interesting as showing an average of the composition of beers manufactured in various countries. It is taken from a very large number of collated analyses.

Average of the content of alcohol, extract, and ash in various beers for export and preservation.

	Per cent. alcohol by volume.			Per cent. extract.			Per cent. ash.		
	Min.	Max.	Mean.	Min.	Max.	Mean.	Min.	Max.	Mean.
<i>French beers.</i>									
Strasbourg.....	4.0	5.8	4.7	4.0	5.6	4.65	0.30	0.35	0.32
Lille.....	4.0	4.2	4.1	4.0	5.3	4.65	0.35
Paris.....	3.5	3.5	3.5	4.0	8.0	6.00
Nancy, Tourtel, Tartonville, Vittel, Vezelise, Toul.	5.0	6.0	5.6	4.0	7.6	5.70	0.19	0.35	0.29
Lyon.....	5.0	5.0
<i>German beers.</i>									
Saxony.....	2.08	6.90	3.70	4.40	7.4	5.8	0.18	0.45	0.25
Bavaria.....	3.00	8.30	4.50	3.90	11.3	7.2	0.13	0.47	0.29
Hanover, Holstein, Pomerania.....	3.93	4.81	4.20	5.07	6.7	5.9	0.25	0.26	0.25

¹ Documents sur les falsifications des matières alimentaires, et sur les travaux du laboratoire municipal, deuxième rapport, Paris, 1885.



Average of the contents of alcohol, extract, and ash in various beers, &c.—Continued.

	Per cent. alcohol by volume.			Per cent. extract.			Per cent. ash.		
	Min.	Max.	Mean.	Min.	Max.	Mean.	Min.	Max.	Mean.
<i>Austrian beers.</i>									
Vienna, Moravia	3.00	4.50	3.5	5.00	8.0	6.1	0.18	0.28	0.20
Bohemia	3.29	4.59	3.6	4.10	5.9	4.7	0.17	0.28	0.20
<i>English beers.</i>									
Ale.....	5.0	8.5	6.2	4.8	14.0	6.6
Porter.....	4.0	6.9	6.4	5.9	7.4	6.5
<i>Belgian beers.</i>									
Lambick	4.5	7.7	6.02	2.07	5.6	3.7	0.30	0.35	0.32
Faro	2.5	4.9	4.15	2.90	5.1	4.2	0.29
Bière d'orge	3.0	4.9	4.35	2.70	4.5	3.4	0.29
Uytzet	2.7	3.2	3.00	4.00	5.0	4.4
Bières blanches	2.2	4.4	3.00	5.0	4.0
Bières diverses	3.5	8.4	5.80	3.10	8.0	5.5

COMPOSITION OF AMERICAN BEER.

But very little work has been done on American beers; they seem to have shared with other dietary articles the general indifference of the American public to the composition of their food and drink.

A very extensive series of analyses was made in the State of New York in 1885, under the authority of the State Board of Health, by Dr. F. E. Englehardt, and outside of this I have been able to find very few published analyses of American beers.

Dr. Englehardt's analyses were made upon a very large number of samples, 476 in all, which were collected from all over the State, and were intended to furnish a good average representation of the beer retailed in the State. The samples included various kinds of malt liquor, porters, ales, and a weak beer sold under the name of weiss beer. Unfortunately no arrangement of the analyses was made with a view to showing the composition of various kinds, as the examination was made principally with reference to the adulteration, so all varieties are tabulated together. The following averages I have had compiled from his table by the Statistical Division of this Department, only excepting a few samples which he has indicated as being imported:

Average composition of American malt liquors, as shown by analyses made for New York State Board of Health by F. E. Englehardt, Ph. D.

Kind.	Specific gravity.	Alcohol by weight.	Extract.	Ash.	Phosphoric acid.
		<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Lager, 172 samples	1.016	3.754	5.864	.259	.0964
Ale, 199 samples	1.013	4.622	5.423	.307	.0832
Porter, 70 samples	1.015	4.462	6.003	.345	.0942
Weiss, 28 samples	1.006	1.732	2.356	.189	.0491

The maximum and minimum content of alcohol, extract, and ash in the same samples is as follows :

Kind.	Maximum.			Minimum.		
	Alcohol by weight.	Extract.	Ash.	Alcohol by weight.	Extract.	Ash.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Lager	7.061	9.647	.412	.677	3.655	.172
Alé.....	8.994	9.501	.552	2.410	2.703	.197
Porter....	6.695	11.783	.557	1.671	2.843	.170
Weiss.....	3.179	4.143	.468	.755	1.277	.069

These analyses show great lack of uniformity of composition in the different varieties of malt liquor, but it should be remembered that the samples were collected with a view to ascertaining the extent of adulteration, and many samples were found to be sophisticated in one way or another. Especially in the case of the content of ash the average of these samples does not give the average composition of American beers, for many of these ashes were found to consist principally of salt.

Following is the average of nineteen analyses made by the same chemist for the New York Board of Health in 1882 :

Specific gravity.....	1.0162
Alcohol by weight.....	2.78
Extractive matter.....	6.047
Sugar.....	1.521
Free acid calculated as lactic....	.189
Ash.....	.305
Phosphoric acid.....	.105

The following analyses of four samples of beer sold in Indianapolis, Ind., were made by Mr. J. N. Hurty :¹

	Specific gravity.	Extract.	Sugar.	Dextrin.	Albuminoids.	Bitter matter.	Acidity calculated as acetic.	Alcohol.
Milwaukee	1.0174	7.312	1.895	3.880	.037	1.530	.159	5.350
Lieber's	1.0229	5.988	3.126	2.644	.016	.202	.281	9.990
Maus's	1.0180	6.330	4.060	2.060	.006	.118	.309	5.384
Schmidt's	1.0172	5.816	3.440	2.283	.014	.074	.080	4.640
Average ...	1.0189	6.361	3.130	2.719	.018	.481	.207	6.341

The analyst does not state whether the percentage of alcohol is by weight or volume, but on account of its being so high, presumably it is the latter.

¹Analyst, Vol. 7, p. 22.

Analyses made in 1873 of New York beers for the "Moderation Society" by Professor Doremus.¹

Specific grav- ity.	Alcohol.	Extrac- tive matter.	Water.	Non-nitrogen- ized.	Albuminoids.	Ash.	Lactic acid.	Phosphoric acid.	Sugar.	Adulterations or deleteri- ous matter.		
	<i>Pr. ct.</i>	<i>Pr. ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per cent.</i>	<i>Per cent.</i>			
1. 0280	4. 00	8. 5215	87. 40	7. 1605	0. 8750	0. 2970	0. 1890	0. 1300	2. 2105	None.		
1. 0315	2. 00	8. 4580	89. 50	7. 0695	0. 8750	0. 3425	0. 1710	0. 1610	2. 2034	None.		
1. 0175	4. 60	6. 5570	88. 80	5. 4080	0. 7000	0. 3680	0. 0810	0. 1775	1. 2285	None.		
1. 0275	2. 80	8. 3410	88. 80	7. 3530	0. 6125	0. 2675	0. 1080	0. 0575	2. 7826	None.		
1. 0330	3. 40	86. 60	8. 9035	0. 6300	0. 3740	0. 0900	0. 1200	3. 0250	None.		
1. 0210	4. 60	6. 8280	88. 50	5. 7170	0. 6375	0. 3385	0. 1350	0. 1875	2. 6472	None.		
1. 0250	2. 50	6. 9740	90. 40	5. 8565	0. 6650	0. 2905	0. 1620	0. 0875	0. 9726	None.		
1. 0180	2. 80	6. 8600	90. 30	5. 6705	0. 6375	0. 4620	0. 1625	0. 0625	1. 0338	None.		
								Phos. acid.	Malt- ose.	Dex- trose.	Dex- trine.	
1. 0150	3. 10	5. 1840	91. 60	4. 0960	1. 6120	0. 2500	0. 2250	0. 1000	0. 5470	0. 5090	2. 6260	None.
1. 0125	5. 20	5. 4660	89. 20	4. 1940	0. 7870	0. 2590	0. 2250	0. 1000	0. 3120	1. 1220	2. 3010	None.
1. 0155	4. 30	6. 0400	89. 60	4. 6870	0. 8400	0. 2410	0. 2700	0. 1120	0. 2040	1. 0580	2. 6980	None.
1. 0120	5. 20	5. 0740	89. 60	3. 7620	0. 7700	0. 2720	0. 2700	0. 1170	0. 7540	0. 3910	1. 9670	None.
1. 0150	4. 60	6. 4360	88. 80	5. 6680	0. 4550	0. 2100	0. 1620	0. 0850	0. 6890	0. 8450	3. 5760	None.
1. 0150	4. 60	6. 1680	89. 20	4. 7000	0. 8570	0. 2950	0. 3150	0. 1200	0. 0950	0. 2060	2. 3750	None.

Analyses made by Professor Englehardt for same society.

Specific grav- ity.	Alcohol.	Extrac- tive or solid matter.	Water.	Maltose- sugar.	Dextrose.	Albuminoids.	Lactic acid.	Ash.	Phosphoric acid.	Adulterations or deleteri- ous matter.
	<i>Per cent.</i>		<i>Per cent.</i>							
1. 0145	4. 25	5. 750	90. 00	1. 420	2. 745	0. 599	0. 153	0. 302	0. 093	None.
1. 0150	3. 70	5. 670	90. 60	1. 426	2. 680	0. 677	0. 174	0. 279	0. 107	None.
1. 0156	3. 70	5. 770	90. 50	1. 590	2. 510	0. 706	0. 150	0. 309	0. 107	None.
1. 0134	4. 25	5. 350	90. 30	1. 285	2. 563	0. 319	0. 212	0. 319	0. 318	None.
1. 0197	3. 50	6. 470	89. 90	1. 434	3. 159	0. 809	0. 150	0. 321	0. 076	None.
1. 0187	3. 70	6. 462	89. 70	1. 563	3. 303	0. 760	0. 202	0. 311	0. 078	None.
1. 0120	4. 10	4. 297	90. 60	0. 913	2. 037	0. 624	0. 150	0. 321	0. 076	None.
1. 0175	4. 30	6. 586	89. 00	1. 435	3. 141	0. 657	0. 123	0. 337	0. 097	None.
1. 0174	4. 20	6. 380	89. 40	1. 448	3. 004	0. 810	0. 212	0. 339	0. 098	None.
1. 0162	4. 25	6. 209	89. 50	1. 485	3. 033	0. 700	0. 212	0. 352	0. 098	None.

The tables furnished above constitute about all the analyses of American beer I have been able to find in the literature I have access to. Probably more have been published in the trade journals.

ANALYSES OF BEERS BY THE UNITED STATES DEPARTMENT OF AGRICULTURE.

The analyses made by this Department comprise 32 samples, this being about all the different brands and varieties of beers of domestic manufacture obtainable in Washington. The investigation was made principally with a view to ascertain the extent and nature of their adulteration, if any, and especially the use of antiseptic and preservative agents. As a basis for determining adulteration, however, it is necessary to know the normal or average composition, so a fairly com-

¹ Am. Analyst, April 1, 1887.

plete analysis of all samples examined has been made. The intention of the investigation was not so much to make a very extensive series of analyses as to establish definite methods of analysis for the guidance of analysts of state boards of health or similar bodies, whose province it is more especially to investigate the extent of adulteration prevailing in their States by the examination of large numbers of samples.

SAMPLES.

The malt liquors used as samples were all purchased in Washington, D. C., and included the various popular brands made in Milwaukee, Cincinnati, Philadelphia, New York, &c., which are sold all over the country, as well as the product of the few local brewers. Some were obtained from wholesale dealers, but the majority were purchased in retail saloons and groceries, without statement of the purpose for which they were intended. All the draft beers were obtained in this way.

A few English and German beers and ales were analyzed for comparison.

Analyses of malt liquors by United States Department of Agriculture.

Designation.	Manufactured in—	Serial number.	Number of analyses.	Specific gravity.	Alcohol by weight.	Alcohol by volume.	Extract.	Original gravity.	Ash.	Reducing sugar as maltose.	Dextrin.	Albuminoids.	Free acids as lactic.	Phosphoric acid.	Carbonic acid.	Remarks.
				<i>Pr. ct.</i>	<i>Pr. ct.</i>	<i>Pr. ct.</i>	<i>Pr. ct.</i>	<i>Pr. ct.</i>	<i>Pr. ct.</i>	<i>Pr. ct.</i>	<i>Pr. ct.</i>	<i>Pr. ct.</i>	<i>Pr. ct.</i>	<i>Pr. ct.</i>	<i>Pr. ct.</i>	
Lager beer, bottled	Milwaukee, Wis.	4800	1	1.0100	4.28	5.39	4.18	1.0505	.196	1.10	1.57	.511	.057	.065	.411	
Export beer, bottled	do	4801	2	1.0140	4.42	5.55	5.40	1.0537	.309	1.06	1.06	.400	.057	.056	.300	Salicylated.
Lager beer, bottled	Alexandria, Va.	4802	3	1.0171	4.55	5.71	5.71	1.0607	.355	2.04	2.21	.681	.074	.091	.489	Salicylated.
Do.	Washington, D. C.	4803	4	1.0143	4.18	5.24	5.05	1.0533	.388	1.25	0.98	.669	.059	.086	.415	
Do.	Cincinnati, Ohio	4804	5	1.0100	5.63	6.94	4.55	1.0628	.240	0.94	2.25	.513	.073	.082	.328	
Export beer, bottled	Saint Louis, Mo.	4805	6	1.0178	4.40	5.47	6.15	1.0500	.312	2.14	2.54	.463	.067	.074	.471	Salicylated.
Lager beer, bottled	Philadelphia, Pa.	4806	7	1.0147	4.29	5.39	5.22	1.0549	.241	1.46	2.30	.738	.078	.071	.717	Salicylated and soured.
Do.	do	4807	8	1.0147	4.35	5.47	5.09	1.0549	.272	1.37	1.80	.738	.080	.104	.219	
"Budweiss" beer, bottled	do	4808	9	1.0141	4.32	5.63	5.94	1.0609	.241	1.46	2.57	.531	.086	.078	.324	
Lager beer, draft	Buffalo, N. Y.	4810	10	1.0241	3.84	4.78	7.05	1.0601	.222	2.81	3.09	.519	.035	.069	
Do.	Philadelphia, Pa.	4811	11	1.0132	4.36	5.47	4.63	1.0539	.265	1.17	1.82	.636	.046	.095	Substitutes for hops used.
Do.	Washington, D. C.	4812	12	1.0146	4.29	5.39	5.18	1.0545	.236	1.22	2.21	.669	.044	.086	
Do.	Cincinnati, Ohio	4813	13	1.0169	4.63	5.78	5.86	1.0607	.235	2.37	2.29	.456	.074	.085	
Do.	Alexandria, Va.	4814	14	1.0137	4.71	5.86	4.91	1.0585	.263	1.10	2.40	.619	.008	.089	Bicarbonate of soda used.
Do.	Washington, D. C.	4815	15	1.0140	4.30	5.39	4.83	1.0538	.262	1.49	1.45	.681	.071	.087	Sulphite used.
Pale lager beer, bottled	Saint Louis, Mo.	4816	16	1.0181	3.86	4.85	5.62	1.0527	.183	1.62	2.59	.619	.000	.083	Bicarbonate of soda added.
"Erlanger" beer, bottled	do	4817	17	1.0178	4.28	5.39	4.61	1.0527	.183	1.62	2.59	.619	.000	.083	Salicylated.
Ale, bottled	Philadelphia, Pa.	4818	18	1.0203	4.68	5.86	6.82	1.0630	.212	2.51	2.58	.463	.046	.093	.344	
Bass pale ale, bottled	England	4819	19	1.0059	6.24	7.74	3.46	1.0647	.401	0.59	0.90	.531	.232	.085	.503	
English porter, bottled	do	4820	20	1.0095	5.66	7.09	4.42	1.0633	.309	0.49	2.20	.500	.117	.056	
Lager beer, bottled	Boston, Mass.	4821	21	1.0147	5.30	7.66	5.90	1.0728	.371	0.57	2.76	.763	.151	.049	.397	
"Kaiser" beer, bottled	Bremen	4822	22	1.0077	5.30	6.63	3.94	1.0587	.328	1.06	1.63	.556	.107	.065	Salicylated.
"Verzandt" beer, bottled	Bavaria	4823	23	1.0036	5.38	6.71	3.05	1.0543	.162	0.69	1.36	.263	.089	.045	
Export beer, bottled	Milwaukee, Wis.	4824	24	1.0197	3.86	4.85	6.24	1.0553	.190	1.71	3.32	.419	.085	.073	Salicylated.
Ale, draft	Philadelphia, Pa.	4825	25	1.0150	4.59	5.71	5.38	1.0581	.194	1.87	2.46	.425	.071	.059	
Ale, bottled	do	4826	26	1.0171	5.25	6.55	6.02	1.0669	.331	1.40	2.80	.569	.094	.057	
Porter, bottled	Reading, Pa.	4827	27	1.0125	6.92	8.63	5.55	1.0781	.472	0.92	1.99	.731	.382	.077	.441	
Do.	do	4828	28	1.0269	4.89	6.10	8.19	1.0781	.472	0.92	1.99	.731	.382	.077	.441	
"Select" beer, bottled	Milwaukee, Wis.	4829	29	1.0183	4.22	5.32	3.88	1.0756	.412	2.67	2.88	.763	.166	.100	.592	
Export beer, bottled	do	4830	30	1.0183	4.22	5.32	3.88	1.0756	.412	2.67	2.88	.763	.166	.100	.592	
"Bohemian" beer, bottled	do	4843	31	1.0183	4.22	5.32	3.88	1.0567	.223	1.82	3.12	.413	.053	.058	.242	
"Bavarian" beer, bottled	do	4844	32	1.0187	4.16	5.24	5.88	1.0563	.224	1.75	3.04	.406	.071	.057	
do	do	4845	32	1.0187	5.06	6.32	6.26	1.0660	.346	1.75	2.87	.556	.074	.077	.265	
Average (28 samples) ¹			1.0161	4.63	5.79	5.33	1.0597	.279	1.65	2.33	.563	.082	.077	.398	

¹ In the averages the five samples of foreign beer were omitted.

METHODS OF ANALYSIS.

In the work on malt liquors an endeavor has been made to simplify the analyses as much as possible, and various methods have been tried with this end in view. The various processes given are believed to be the best possible for combining rapidity of execution with sufficient accuracy of results.

The necessary determinations may be conveniently divided into two classes :

1. The analysis of the sample proper, comprising determination of the density, alcohol, extract or total solids, original gravity, saccharine matter, albuminoids, free acid (fixed), free acid (volatile), ash, glycerine, phosphoric acid, and carbonic acid.

2. The processes for the detection of adulteration, comprising a search for substitutes for malt, substitutes for hops, preserving agents (salicylic acid, borax, sulphites), and mineral additions.

ALCOHOL.

The estimation of alcohol in beers and wines is generally made in one of two ways, either by direct distillation and determining the alcohol in the distillate, or, indirectly, by evaporating the alcohol from a sample and obtaining the per cent. from the difference in specific gravity of the sample before and after the alcohol has been driven off. Authorities differ as to the accuracy of the indirect method, some even holding it to give better results than the direct estimation. It is generally recommended to use both methods, as the one gives a check over the other, and it is very easy to carry both on together, as the same sample which is used for the distillation can be used for the determination of the density of the de-alcoholized solution, provided no tannin is used. I much prefer the distillation method, and have adopted the results by it in the tables.

I have almost invariably found that during the distillation a precipitation of flocculent albuminous matter takes place in the flask, evidently bodies which are rendered insoluble, either by the evaporation of the alcohol or acetic acid, or by the heating of the solution, and it would seem obvious that this separation of solids from the solution would vitiate more or less the results by the indirect method.

For the distillation method 100cc. of the sample, freed from carbonic acid by shaking, are measured out, rinsed into a flask with about 50cc. water, the latter connected with a Liebig's condenser, and 100cc. distilled off. The sample and distillate should be measured at the same temperature. The specific gravity of the distillate is then obtained by means of an accurately tared pycnometer, preferably one carrying a thermometer, so that the weight may be taken at exactly 15.5° C. The per cent. of alcohol in the distillate is then obtained by reference to a table giving the per cent. of alcohol in solutions of different specific

gravities, of which tables Hehner's are the best in use. This per cent. multiplied by the specific gravity of the distillate, and the result divided by the specific gravity of the original sample, gives the per cent. of alcohol by weight contained in the latter. The accuracy of the operation is increased by weighing the sample taken, and also the distillate. Then the weight of the distillate multiplied by the per cent. of alcohol corresponding to its specific gravity as found in the table, and the result divided by the weight of the sample taken, gives the per cent. of alcohol by weight contained in the latter.

For the indirect method it is necessary to estimate accurately the specific gravity of the original beer, thoroughly freed of carbonic acid by shaking and standing; then 100cc. or any convenient quantity is measured out, evaporated to half its bulk, cooled, and made up to its original volume with water, taking care to have the solution at the same temperature as the sample when first measured. The specific gravity of the de-alcoholized liquor is taken. Then the specific gravity of the original sample divided by the specific gravity of the de-alcoholized solution gives the specific gravity of the alcohol evaporated, from which figure the per cent. of alcohol is ascertained by reference to the table. The same sample which is used for distilling can be very conveniently used for this determination as well.

Hegner's alcohol table.

Specific grav- ity at 15.5°C.	Alcohol by weight.	Alcohol by volume.	Specific grav- ity at 15.5°C.	Alcohol by weight.	Alcohol by volume.	Specific grav- ity at 15.5°C.	Alcohol by weight.	Alcohol by volume.	Specific grav- ity at 15.5°C.	Alcohol by weight.	Alcohol by volume.
1.0000	<i>Per ct.</i> 0.00	<i>Per ct.</i> 0.00		<i>Per ct.</i> 4.06	<i>Per ct.</i> 5.08		<i>Per ct.</i> 8.71	<i>Per ct.</i> 10.82		<i>Per ct.</i> 14.00	<i>Per ct.</i> 17.26
0.9999	0.05	0.07	0.9929	4.12	5.16	0.9859	8.79	10.91	0.9789	14.09	17.37
8	0.11	0.13	8	4.19	5.24	8	8.86	11.00	8	14.18	17.48
7	0.16	0.20	7	4.25	5.32	7	8.93	11.08	7	14.27	17.59
6	0.21	0.26	6	4.31	5.39	6	9.00	11.17	6	14.36	17.70
5	0.26	0.33	5	4.37	5.47	5	9.07	11.26	5	14.45	17.81
4	0.32	0.40	4	4.44	5.55	4	9.14	11.35	4	14.55	17.92
3	0.37	0.46	3	4.50	5.63	3	9.21	11.44	3	14.64	18.03
2	0.42	0.53	2	4.56	5.71	2	9.29	11.52	2	14.73	18.14
1	0.47	0.60	1	4.62	5.78	1	9.36	11.61	1	14.82	18.25
0	0.53	0.66	0			0			0		
0.9989	0.58	0.73	0.9919	4.69	5.85	0.9849	9.43	11.70	0.9779	14.91	18.36
8	0.63	0.79	8	4.75	5.94	8	9.50	11.79	8	15.00	18.48
7	0.68	0.86	7	4.81	6.02	7	9.57	11.87	7	15.04	18.58
6	0.74	0.93	6	4.87	6.10	6	9.64	11.96	6	15.17	18.68
5	0.79	0.99	5	4.94	6.17	5	9.71	12.05	5	15.25	18.78
4	0.84	1.06	4	5.00	6.24	4	9.79	12.13	4	15.33	18.88
3	0.89	1.13	3	5.06	6.32	3	9.86	12.22	3	15.42	18.98
2	0.95	1.19	2	5.12	6.40	2	9.93	12.31	2	15.50	19.08
1	1.00	1.26	1	5.19	6.48	1	10.00	12.40	1	15.58	19.18
0	1.06	1.34	0	5.25	6.55	0	10.08	12.49	0	15.67	19.28
0.9979	1.12	1.42	0.9909	5.31	6.63	0.9839	10.15	12.58	0.9769	15.75	19.39
8	1.19	1.49	8	5.37	6.71	8	10.23	12.68	8	15.83	19.49
7	1.25	1.57	7	5.44	6.78	7	10.31	12.77	7	15.92	19.59
6	1.31	1.65	6	5.50	6.86	6	10.38	12.87	6	16.00	19.68
5	1.37	1.73	5	5.56	6.94	5	10.46	12.96	5	16.08	19.78
4	1.44	1.81	4	5.62	7.01	4	10.54	13.05	4	16.15	19.87
3	1.50	1.88	3	5.69	7.09	3	10.62	13.15	3	16.23	19.96
2	1.56	1.96	2	5.75	7.17	2	10.69	13.24	2	16.31	20.06
1	1.62	2.04	1	5.81	7.25	1	10.77	13.34	1	16.38	20.15
0	1.69	2.12	0	5.87	7.32	0	10.85	13.43	0	16.46	20.24
0.9969	1.75	2.20	0.9899	5.94	7.40	0.9829	10.92	13.52	0.9755	16.85	20.71
8	1.81	2.27	8	6.00	7.48	8	11.00	13.62	8	16.92	20.81
7	1.87	2.35	7	6.07	7.57	7	11.08	13.71	7	17.00	20.91
6	1.94	2.43	6	6.14	7.66	6	11.15	13.81	6	17.08	21.01
5	2.00	2.51	5	6.21	7.74	5	11.23	13.90	5	17.16	21.11
4	2.06	2.58	4	6.28	7.83	4	11.31	13.99	4	17.24	21.21
3	2.11	2.62	3	6.36	7.92	3	11.38	14.09	3	17.32	21.31
2	2.17	2.72	2	6.43	8.01	2	11.46	14.18	2	17.40	21.41
1	2.22	2.79	1	6.50	8.10	1	11.54	14.27	1	17.48	21.51
0	2.28	2.86	0	6.57	8.18	0	11.62	14.37	0	17.56	21.61
0.9959	2.33	2.93	0.9889	6.64	8.27	0.9819	11.69	14.46			
8	2.39	3.00	8	6.71	8.36	8	11.77	14.56			
7	2.44	3.07	7	6.78	8.45	7	11.85	14.65			
6	2.50	3.14	6	6.86	8.54	6	11.92	14.74			
5	2.56	3.21	5	6.93	8.63	5	12.00	14.84			
4	2.61	3.28	4	7.00	8.72	4	12.08	14.93			
3	2.67	3.35	3	7.07	8.80	3	12.15	15.02			
2	2.72	3.42	2	7.13	8.88	2	12.23	15.12			
1	2.78	3.49	1	7.20	8.96	1	12.31	15.21			
0	2.83	3.55	0	7.27	9.04	0	12.38	15.30			
0.9949	2.89	3.62	0.9879	7.33	9.13	0.9809	12.46	15.40			
8	2.94	3.69	8	7.40	9.21	8	12.54	15.49			
7	3.00	3.76	7	7.47	9.29	7	12.62	15.58			
6	3.06	3.83	6	7.53	9.37	6	12.69	15.68			
5	3.12	3.90	5	7.60	9.45	5	12.77	15.77			
4	3.18	3.98	4	7.67	9.54	4	12.85	15.86			
3	3.24	4.05	3	7.73	9.62	3	12.92	15.96			
2	3.29	4.12	2	7.80	9.70	2	13.00	16.05			
1	3.35	4.20	1	7.87	9.78	1	13.08	16.15			
0	3.41	4.27	0	7.93	9.86	0	13.15	16.24			
0.9939	3.47	4.34	0.9869	8.00	9.95	0.9799	13.23	16.33			
8	3.53	4.42	8	8.07	10.03	8	13.31	16.43			
7	3.59	4.49	7	8.14	10.12	7	13.38	16.52			
6	3.65	4.56	6	8.21	10.21	6	13.46	16.61			
5	3.71	4.63	5	8.29	10.30	5	13.54	16.70			
4	3.76	4.71	4	8.36	10.38	4	13.62	16.80			
3	3.82	4.78	3	8.43	10.47	3	13.69	16.89			
2	3.88	4.85	2	8.50	10.56	2	13.77	16.98			
1	3.94	4.93	1	8.57	10.65	1	13.85	17.08			
0	4.00	5.00	0	8.64	10.73	0	13.92	17.17			

EXTRACT OR TOTAL SOLIDS.

This can also be obtained either directly, by weighing or measuring a sample into an open dish, driving off the moisture at 100° C. until a constant weight is obtained, or indirectly, by calculation from the specific gravity of the de-alcoholized solution. For the direct estimation a small quantity should be weighed out; 10 grams is quite sufficient, and 5 grams gives still better results. This is allowed to run out into a thin film on the bottom of a shallow dish having an area of several square inches. In this shape it is very readily dried to a constant weight at 100° C., while if a larger quantity is taken this becomes quite difficult, and it is necessary to use a higher temperature.¹

In case the indirect method is used the per cent. of extract may be obtained from one of the various tables in use or by dividing the difference between the specific gravity and 1.000 by the factor 3.86. The result obtained is the number of grams of solid matter in 100cc. of the beer, and must be divided by the specific gravity of the original beer to get the exact per cent.

ORIGINAL GRAVITY.

The "original gravity" of beer is the specific gravity of the wort from which it was made, before fermentation. This is ascertained by computation from data given by the alcoholic content and the malt extract contained in the de-alcoholized liquid. The specific gravity of the alcoholic distillate (or the specific gravity of the alcohol evaporated, if the indirect method is used) when subtracted from 1.000 gives a number called the "spirit indication." The degrees of gravity lost are then ascertained by reference to the table given below. The degrees found are added to the specific gravity of the de-alcoholized beer, and the number thus obtained is the original gravity.

The following table was calculated by Graham, Hofmann, and Redwood from actual experiments on malt worts fermented under normal conditions:

¹ The following duplicate determinations show very satisfactory agreement in the results obtained by using a small quantity for drying:

	No. 4800.	No. 4801.	No. 4804.	No. 4817.
1.....	4.25	5.40	4.52	4.67
2.....	4.16	5.40	4.58	4.62

Degrees of spirit indication.	.0	.1	.2	.3	.4	.5	.6	.7	.8	.9
03	.6	.9	1.2	1.5	1.8	2.1	2.4	2.7
1	3.0	3.3	3.7	4.1	4.4	4.8	5.1	5.5	5.9	6.2
2	6.6	7.0	7.4	7.8	8.2	8.6	9.0	9.4	9.8	10.2
3	10.7	11.1	11.5	12.0	12.4	12.9	13.3	13.8	14.2	14.7
4	15.1	15.5	16.0	16.4	16.8	17.3	17.7	18.2	18.6	19.1
5	19.5	19.9	20.4	20.9	21.3	21.8	22.2	22.7	23.1	23.6
6	24.1	24.6	25.0	25.5	26.0	26.4	26.9	27.4	27.8	28.3
7	28.8	29.2	29.7	30.2	30.7	31.2	31.7	32.2	32.7	33.2
8	33.7	34.3	34.8	35.4	35.9	36.5	37.0	37.5	38.0	38.6
9	39.1	39.7	40.2	40.7	41.2	41.7	42.2	42.7	43.2	43.7
10	44.2	44.7	45.1	45.6	46.0	46.5	47.0	47.5	48.0	48.5
11	49.0	49.6	50.1	50.6	51.2	51.7	52.2	52.7	53.3	53.8
12	54.3	54.9	55.4	55.9	56.4	56.9	57.4	57.9	58.4	58.9
13	59.4	60.0	60.5	61.1	61.6	62.2	62.7	63.3	63.8	64.3
14	64.8	65.4	65.9	66.5	67.1	67.6	68.2	68.7	69.3	69.9
15	70.5	71.1	71.7	72.3	72.9	73.5	74.1	74.4	75.3	75.9

A content of acetic acid above 0.1 per cent. somewhat vitiates the results given by the table, and a correction is necessary. The manner of making this correction may be ascertained by consulting Allen,¹ or any of the standard works, which give also tables for the calculation of the actual weight of malt used in the wort.

Below is given a table showing the results obtained from the samples analyzed, by the direct and indirect methods :

Comparison of direct and indirect methods of estimating alcohol and extract in malt liquors.

Serial No.	Number of analysis.	Alcohol direct.	Alcohol by calculation.	Extract direct.	Extract by calculation.	Original gravity by distillation.	Original gravity by evaporation.
		<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>		
4800.....	1	4.28	4.33	4.18	4.56	1.0505	1.0506
4801.....	2	4.42	5.40	6.03	1.0573
4802.....	3	4.55	4.51	5.71	6.44	1.0607	1.0596
4803.....	4	4.18	3.96	5.05	5.52	1.0533	1.0516
4804.....	5	5.53	5.39	4.55	5.03	1.0628	1.0616
4805.....	6	4.40	4.08	6.15	6.44	1.0590	1.0562
4806.....	7	4.29	4.08	5.22	5.67	1.0549	1.0531
4807.....	8	4.35	3.84	5.09	5.54	1.0549	1.0507
4808.....	9	4.52	4.39	5.94	6.64	1.0609	1.0594
4810.....	10	3.84	3.67	7.05	7.82	1.0601	1.0584
4811.....	11	4.36	4.08	4.63	5.29	1.0539	1.0516
4812.....	12	4.29	3.90	5.18	5.57	1.0545	1.0513
4813.....	13	4.63	4.33	5.86	6.32	1.0607	1.0576
4814.....	14	4.71	4.57	4.91	5.62	1.0585	1.0569
4815.....	15	4.30	3.90	4.83	5.39	1.0538	1.0506
4816.....	16	3.86	5.62
4817.....	17	4.28	3.90	4.64	6.36	1.0577	1.0545
4818.....	18	4.68	4.33	6.82	7.24	1.0650	1.0613
4819.....	19	6.24	5.83	3.46	4.17	1.0647	1.0616
4820.....	20	5.66	5.39	4.42	4.90	1.0633	1.0611
4821.....	21	6.13	5.77	5.90	6.33	1.0728	1.0697
4822.....	22	5.30	5.51	3.94	4.54	1.0587	1.0605
4823.....	23	5.38	5.07	3.05	3.25	1.0543	1.0520
4824.....	24	3.86	3.43	6.24	6.63	1.0553	1.0517
4825.....	25	4.59	4.20	5.38	5.80	1.0581	1.0545
4826.....	26	5.25	4.76	6.02	6.55	1.0669	1.0623
4827.....	27	6.92	6.42	5.55	6.09	1.0781	1.0741
4828.....	28	4.89	4.39	8.19	8.83	1.0736	1.0683
4842.....	29	4.22	3.49	5.88	6.31	1.0570	1.0509
4843.....	30	4.22	3.37	5.84	6.23	1.0567	1.0493
4844.....	31	4.16	3.37	5.88	6.26	1.0563	1.0494
4845.....	32	5.06	4.14	6.26	6.69	1.0660	1.0576
Average	4.73	4.41	5.40	5.94	1.0600	1.0569

¹ Commercial Organic Analysis, 2d edit., Vol. I.

SACCHARINE MATTER.

The saccharine matter in beer consists principally of maltose and dextrin, with probably a small proportion of dextrose. The greater part of the entire extract is composed of these different carbohydrates. The proportion of maltose to dextrin is of considerable importance in judging of the quality of a beer. The dextrin contributes to the "fullness" (*vollmündigkeit*) of the taste, and a larger proportion of dextrin to extract makes a beer of good "body." The method of brewing is said to have an important influence upon the relative proportions of maltose and dextrin. The determination of the percentages of these sugars is, therefore, quite an important one as showing the nature and quality of the sample, though not of much utility in detecting adulterations. The usual method is to estimate the maltose by Fehling's solution, and the dextrin, if it is reported at all, is obtained by difference from the total solids. So little is known of the saccharine bodies already existing in the grain and of the products of the conversion of starch into sugars, that their separate estimation is rather unsatisfactory.¹ The dextrin may be determined directly by precipitating with alcohol, washing and weighing. The results are sufficiently accurate for commercial purposes, according to J. West Knights,² who employed it upon worts.

Graham estimates the maltose and dextrin in beer worts by the use of Fehling's solution before and after inversion.³ The cupric oxide reducing power is determined gravimetrically, 10cc. are measured out and diluted to 100cc.; 20cc. of this solution are used to 30cc. Fehling's solution. The weight of CuO obtained, multiplied by 0.7314 gives the amount of maltose in the quantity of diluted liquid employed. The maltose having been determined, 10cc. of the wort are mixed with 3cc. of sulphuric acid, diluted to 100cc. and inverted by heating to 100° C., for 3 to 4 hours in a flask furnished with a long tube. The volume of the solution is again made up to 100cc., 10cc. carefully measured or weighed, neutralized with sodium carbonate, and the reducing power determined by heating with Fehling's solution, in the same way as before. The percentage of dextrin is then calculated as follows: Multiply half the weight of CuO, obtained by the action of Fehling's solution on 2cc. of the original wort by 1.72, and subtract the product from the CuO obtained from the inverted solution (= 1cc. of the original). The difference multiplied by 40.8 gives the *grams* of dextrin in 100cc. of the original wort.

This method was applied to the samples analyzed with very unsatisfactory results. In some cases the sum of the maltose and dextrin determined in this way exceeded the amount of total extract, while in other cases the per cent. of dextrin was a minus quantity. This method was therefore abandoned. The results given were obtained by

¹ Recent work on this problem has been published by O'Sullivan, *Jour. Chem. Soc.*, Jan., 1886, p. 53.

² Allen's *Organic Analysis* 1, 274.

³ *Analyst* 7, 211.

the following method taken from Allen.¹ The maltose was estimated by Fehling's solution, volumetrically, by the method in use in this laboratory for the determination of cupric oxide reducing power.² The polarization is then observed in the original beer, the clarification being accomplished by means of lead acetate. The reading is taken on the circular scale, and constitutes the total circular rotation. The number of grams of maltose in 100cc. of the beer having been ascertained from the results obtained with Fehling's solution, it is multiplied by 2.78, which gives the rotation due to maltose; this result is deducted from the total rotation, which gives the rotation due to dextrin. The angle found, divided by 3.86 or multiplied by .259, gives the grams of dextrin in 100cc of the solution. These figures are based on the assumption that the polariscope used is one in which monochromatic light is employed, and that the liquid is observed in a tube 200 millimeters in length. By dividing the grams per 100cc. by the density of the beer, the actual percentage of maltose and dextrin will be ascertained.

As considerable interest is attached to the nature of the polarizing bodies in malt liquors, I append the polarization given by the samples examined. It is a very easy and satisfactory determination to make, the beers being readily clarified by acetate of lead, giving bright, clear solutions. The figures given are in divisions of the cane-sugar scale, and for the normal beer, the dilution of one-tenth incident upon the addition of the lead being corrected by reading in a 220-millimeter tube; the instrument employed was a Laurent polariscope, in which monochromatic light is employed.

Serial number.	Normal polarization.	Serial number.	Normal polarization.
	°		°
4800	42.3	4817	77.6
4801	61.3	4818	79.4
4802	66.6	4819	18.7
4803	33.9	4820	45.8
4804	52.5	4821	57.2
4805	74.4	4822	42.8
4806	60.4	4823	33.1
4807	50.4	4824	82.5
4808	74.8	4825	68.6
4810	93.0	4826	69.7
4811	47.5	4827	47.8
4812	55.8	4828	87.6
4813	72.2	4842	75.4
4814	57.5	4843	78.2
4815	45.5	4844	78.8
4816	65.8	4845	74.8

The polarization was in all cases right-handed.

ALBUMINOID MATTERS.

The albuminoids were determined by weighing 10 grams of the beer into a schälchen, evaporating to dryness, and burning with soda lime in the usual way. The nitrogen found $\times 6.25$ is given as the per cent.

¹ Page 275.

² Fully described in Bulletin No. 15, page 32.

age of albuminoids. Graham determines the nitrogen by "Wanklynizing" the beer as in water analysis. The determination of the nitrogenous matters in beer is important in enabling the analyst to form an opinion on the question whether substitutes for malt were used in its manufacture, as saccharine matter derived from sources other than grain have little nitrogenous content. Too large an amount of albuminous matter is injurious to the keeping qualities of the beer.

The Kjeldahl¹ method, which was originally devised by its inventor for application to the determination of nitrogen in beers and worts, may also be used.

FREE ACIDS.

The ideas of chemists in regard to the nature of the acidity of normal beer have undergone considerable change in the last two or three years. It was formerly considered to be principally due to the presence of lactic acid, with a small quantity of succinic and other acids, but is now considered to be due, for the greater part, to acid phosphates. Acetic acid is present only to a very limited extent in normal beer, its presence in any considerable quantities being proof of the "souring" of the beer. Ott² has shown the difficulty of ascertaining the exact point of neutralization in beer, as by the addition of alkali to the acid phosphates the reaction becomes "amphoteric" from the simultaneous formation of both primary and secondary phosphates, and the establishment of the point of neutralization by the reaction with litmus paper is very difficult. No better means of determining acidity in beer has been proposed, however, and I have used the ordinary method of adding standard alkali until a drop placed on neutral litmus paper produces no alteration of color. 50cc are conveniently taken for this determination, freed from carbonic acid and titrated with decinormal alkali. The acidity can be given directly as cubic centimeters of normal alkali required for 100cc. of beer, or reckoned as lactic acid. The volatile acids, when it is necessary to determine them separately, as in the case of soured beer, may be best estimated by distillation in a current of steam, as described under wine, all methods for their estimation by difference by evaporating the beer to dryness and titrating the residue having been shown to be faulty.

The Bavarian chemists,³ at their last meeting in 1886, adopted the figure of 3cc. normal alkali for 100cc. beer as a maximum limit for a normal beer.

ASH.

The ash may best be determined by burning the residue from 100 cc. of beer at a very low red heat in a muffle. The ash obtained

¹ Zeit. Anal. Chem. 1883, 366, for reference to the modifications of the method, see Bulletin No. 12, U. S. Depart. Agriculture, Division of Chemistry, p. 55.

² Zeit. Anal. Chem. 24, 132.

³ Ber. ü. d. fünfte Ver. der Freien Verein Bay. Ver. d. angewandten Chem. zu Würzburg, Berlin, 1887.

should be subjected to a qualitative examination, with a view to ascertaining if any mineral substances have been added to the beer.

GLYCERINE.

The estimation of glycerine is a troublesome and unsatisfactory determination to make, and as the information obtained in the case of beer is not sufficient to repay the labor of the analysis, except in special cases, I have omitted it.

The following method is used by the Bavarian chemists : 50cc. of beer are treated with about 3 grams of quicklime, evaporated to a sirup, then mixed with about 10 grams coarsely powdered marble or sand, and brought to dryness. The entire dried mass is transferred to an extraction apparatus, and extracted for six or eight hours with not over 50cc. strong alcohol. To the slightly colored extract is added an equal volume of water-free ether, and the solution after standing a short time is poured into a weighed flask, or filtered through a small filter, which is afterwards washed with a little ether-alcohol. After the evaporation of the ether and alcohol the residue is dried in the air bath at 100° to 105° C. in a loosely-closed flask, until the losses in weight are constant. With beers that are very rich in extract, the ash-content of the glycerine may be determined and deducted from the total weight.

The methods recently published for the estimation of glycerine by its conversion into carbonic acid by sulphuric acid and bichromate of potash have been utilized for its determination in fermented liquors by Legler¹ and promise to prove more expeditious and exact than the old methods.

PHOSPHORIC ACID.

The phosphoric acid was determined by means of a standard solution of uranium acetate, except in the case of a few very dark-colored samples, when the analysis was made gravimetrically from the ash by precipitation with ammonium molybdate, in the usual way.

CARBONIC ACID.

Most investigators have given very little attention to the determination of the carbonic acid in beer, regarding it as of little importance in forming an estimate of the quality of the sample examined. The practical consumer, however, is of quite a different opinion and condemns immediately a beer which is "flat" or insufficiently carbonated, however worthy it may be in other respects. The reason for its unimportance as a determination is found in the difficulty of the accurate estimation in the beer as supplied to the customer. The usual method of determining it is to measure or weigh out a convenient quantity of the beer into a flask, connect the latter either with an absorbing apparatus for

¹ Rep. Anal. Chem. 47 Analyst, 1887, 14. See further under wine.

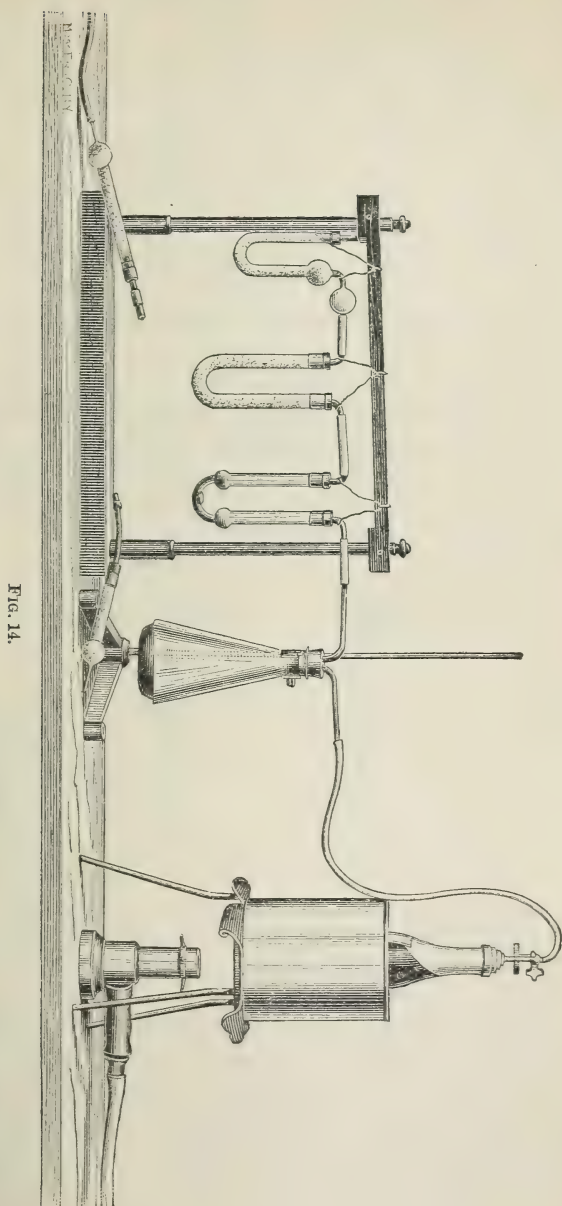
the estimation of the CO_2 direct; or with a suitable apparatus for the retention of water, thus estimating it by loss or indirectly, its liberation from the beer being accomplished by the aid of heat. But this manner of manipulation gives simply the amount of carbonic acid capable of being held in solution by a liquid of the density and temperature of the beer when it was measured out, supposing it to have been fully charged previously. The excess of gas above the saturation point, however, which is held by the beer so long as it is kept under pressure, or at a low temperature, escapes as soon as the pressure is removed and gives the beer its "head," which is so desirable a qualification. This excess of gas soon passes off, but during this short interval the beer is drunk. The problem is to estimate the carbonic acid just as it exists in the beer as it is consumed. This is a difficult matter with beer contained in casks or kegs, though it might be done by drawing the sample off by gas-tight connections as in gas analysis. But where the beer is furnished in stoppered bottles it is an easy matter and furnishes a most valuable index as to the freshness and proper preparation of the beer. Where secondary fermentation or souring has set in, there will be an excess of carbonic acid and the beer will have become cloudy.

Where there is a good content of carbonic acid, but the acidity of the beer is very low, the indications are that bicarbonate of soda has been added.

Hassall speaks of the estimation of CO_2 in bottled aerated waters, the gas being drawn off by means of a champagne tap, and Dr. Wiley¹ has estimated the CO_2 in koumiss in that way, using a calcium chloride tube for retention of the water carried off by the gas, and estimating it by difference, the whole bottle being weighed. In applying this form of apparatus to beer considerable difficulty was experienced on account of the viscosity of the liquid; the bubbles formed were so tenacious that when the bottle was connected directly with a calcium chloride or sulphuric acid tube, the latter would become filled with the beer in a very short time. This difficulty was obviated by the use of the form of apparatus shown in the accompanying figure, devised by Mr. T. C. Trescot and myself.

The cork of the bottle is pierced with a champagne tap, and this is connected with an Erlenmeyer flask, in the broad bottom of which the bubbles are broken and not allowed to pass beyond it; next comes a U tube filled with sulphuric acid, then a calcium chloride tube, then a soda lime tube to absorb the dried CO_2 . The bottle of beer is placed in a convenient vessel—an empty ether can with the tap cut away answers admirably, as shown in the cut—which is nearly filled with cold water. After the apparatus is connected the tap is opened slowly and the gas allowed to flow through the apparatus; when it ceases to flow spontaneously a burner is placed under the can and the tempera-

¹ Am. Chem. Jour. 1886, Ann. Rept. U. S. Dept. Agr'l., 1885, p. 118.



ture gradually raised until it reaches 80° C., beyond which it should not be carried. By holding it at this temperature for about half an hour and taking the bottle out of the can and shaking it occasionally all the CO_2 may be driven off. Then the tube from a suction pump is connected with a calcium chloride tube and this in turn with the soda lime tube. The valve of the tap is then closed, the latter removed from the bottle, connected with a soda lime tube, and by gradually opening the valve, a stream of air, from which the CO_2 has been removed, is drawn through the apparatus by the pump, so that all the CO_2 contained in it is drawn into the soda lime tube and absorbed; the increase in the weight of the latter, of course, gives the weight of CO_2 in the beer. The quantity of the beer is measured or weighed, that which has been carried into the Erlenmeyer flask being added, and the per cent. ascertained.

In the case of large bottles it may be found necessary to add a second soda lime tube, and it is best to use fresh soda lime for each determination. It was found necessary to modify the champagne taps, as the thread with which they are provided cuts the cork too much and allows the escape of the gas. Accordingly this thread was turned off entirely, leaving a smooth tube, as shown in the figure.

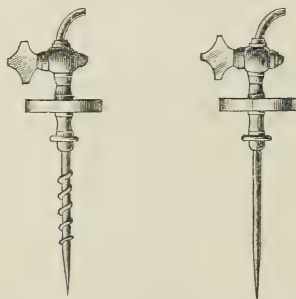


FIG. 15.

This can be forced through the cork with little difficulty, and allows no leakage of gas. It was also found necessary to have the cocks reground to make them fit accurately.

The patent rubber-stopper beer bottles presented a difficulty to this method of analysis, as it was impossible to make a gas-tight connection with them without the escape of the confined gas. In these the gas was estimated by loss of weight, calcium chloride and sulphuric acid tubes and connections being weighed with the bottle on a large balance, the bottle opened and connection made as quickly as possible, the gas driven off as before, and the loss ascertained by weighing the entire apparatus again. It is necessary to raise the heat very gradually to prevent the filling up of the tubes with the beer carried over by the bubbles. There is a slight error from the watery vapor which escapes with the pressure of gas when the bottle is opened, and altogether the determination is not so satisfactory as with the cork-stoppered bottles.

The percentage of CO_2 in the keg beers was not determined. For convenience of inspection the results of the determinations made are given again below :

Number.	Per cent. CO_2 .	Number.	Per cent. CO_2 .
4800	.411	4817	.629
4001	.300	4818	.344
4802	.489	4820	.503
4803	.415	4821	.397
4804	.328	4827	.441
4805	.471	4828	.592
4806	.717	4843	.242
4807	.219	4845	.265
4808	.324		

Nos. 4801–4803 and 4806 had rubber stoppers, the rest cork. No. 4068 was evidently in a state of after-fermentation, cloudy, and acid; excluding that analysis, the 16 others gave an average of .398 per cent. Most authorities give an average of .1 to .2 per cent. in beer. In Nos. 4801 and 4804 duplicate analysis were with different bottles of the same lot, with the following results :

	1.	2.
No. 4801294	.307
No. 4804329	.327

DETECTION OF ADULTERATION.

Probably there is no one article of daily consumption that has been so often subject to suspicion of adulteration or sophistication as beer. Its complex composition and peculiar nature have deceived people into making all sorts of charges against its purity, but experience has failed to establish the truth of by far the greater majority of these charges, and the facts of many published analyses show that it is as free from adulteration as most other articles of consumption, and more so than some. Here comes in the question, so difficult to answer in this country, of what constitutes adulteration or sophistication of an article of food? The definition of what shall constitute a pure malt liquor is hard to settle. Even in Europe, where a much stricter supervision is kept over foodstuffs than here, the definition varies widely. In Bavaria, where more beer per capita is consumed than in any other country, the laws limit the materials from which it is made to barley, malt, hops, yeast, and water, while in England the comprehensive definition has been given to beer as being “a fermented saccharine infusion to which a wholesome bitter has been added.”¹

SUBSTITUTES FOR MALT.

A great deal has been said, pro and con, on the subject of the propriety of the use of other matter than malted barley as a source of

¹ Blyth.

saccharine material for brewing purposes. There may be said to be three ways of substituting saccharine material. First, other grain may be used for malting; second, unmalted starchy matter, that is whole grain, may be added to the malt before it is mashed, the latter being diluted as it were, for the diastase in the malt has converting power sufficient for considerably more starch than is contained in itself; third, the saccharine matter may be supplied already converted, as in commercial starch-sugar, or glucose, cane sugar, inverted cane sugar, &c. Of these different substitutes the third class is probably the more objectionable, as beer brewed from such saccharine matter is lacking in various constituents derived from the grain, which are important additions to its nutritive power, namely, the phosphatic salts and the nitrogenous bodies.

In much the same way would bread made from starch alone be lacking in nutritive value.

There is no way of determining directly or absolutely that a beer has been brewed partially from glucose, but it may be inferred from its small content of those constituents which are contained in malt, but not in glucose, such as phosphoric acid and albuminoids, and the existence in the ash of large proportions of such salts as are known to form a large part of the ash of commercial starch-sugar, as sulphates. König gives .05 per cent. of phosphoric acid as the lowest limit for a beer containing 5 per cent. of extract or over.

The association of Bavarian chemists depends on the estimation of the nitrogen for the detection of the use of malt substitutes, and establishes the minimum of .65 per cent. of nitrogen (4 per cent. of albuminoids) in the extract. It is very evident that these figures are too high for American beers; only two of the samples examined, Nos. 4821 and 4823, contain less than .05 per cent. of phosphoric acid, and these are both imported beers; while the average content of the samples of American beer is .077. Not a single one of the samples contains as low as .65 per cent. of nitrogen in the extract, most of them containing about 1 per cent., while some give over 2 per cent. Dr. Englehardt's samples show a still higher average per cent. of phosphoric acid. Unfortunately there was no determination of the albuminoids in his samples. Yet it is a well-known fact that very few beers are made in this country without more or less malt substitution. Nothing can settle this point and enable the analyst to decide positively whether malt substitutes have been used until a standard is established by the analysis of a large number of samples known to be brewed from pure malt alone.

SUBSTITUTES FOR HOPS.

The nature of the bitters used in beer has long been the target towards which public suspicion is directed, and nearly every substance known possessing a bitter taste has been enumerated among the adulterations of beer, from poisonous alkaloids, such as strychnin and pic-

rotoxin, to harmless or quasi-harmless bitter roots and woods, such as quassia, gentian, &c. Complete and exhaustive schemes of analysis have been compiled, such as Dragendorff's, Ender's, &c., for the detection and isolation of such foreign bitters. Either these methods of investigation are faulty or difficult of manipulation, or the use of foreign bitters is very much less prevalent than is generally supposed, for the cases where such bitters have been detected and isolated are very scarce in chemical literature. In fact, Elsner, a German authority on food adulterations, goes so far as to say that there has never been a case where the existence of a foreign bitter in a malt liquor has been proven with certainty. This is going too far, of course, for picrotoxin and picric acid have undoubtedly been found in beers, and probably more cases of such adulteration would occasionally have been discovered were it not for the difficulty of the analysis and the small quantity of matter required for imparting a bitter taste. But there is probably much less of this hop substitution than the space given it in works on the subject would indicate. Hops not only give the bitterness to beer but also impart to it its peculiar aroma, and enhance its keeping qualities, and unless it were at a time when they were very dear it would hardly pay the brewer to sacrifice the good flavor and keeping qualities of his beer in order to save a few cents a pound in his bitters.

It is stated by authorities on the subject that the bitter matter of hops is precipitated by acetate of lead, while with *all* hop substitutes the filtrate from the lead precipitate retains its bitter taste. The excess of lead should be precipitated by sulphureted hydrogen before the filtrate is tasted for bitterness. I examined qualitatively by this test all the samples analyzed and found them all free from foreign bitters according to it, with one exception, No. 4811, which contained a bitter other than hops, though not in sufficient quantity to admit of its separation and identification. All the samples except Nos. 4801, 4811, and 4815 gave a plainly perceptible odor of hops in the distillate.¹

PRESERVING AGENTS.

We come now to what I consider the most important sophistication of beer at the present day and the most reprehensible and most deserving of repressive legislation. The use of artificial preserving agents not only introduces foreign matters into the beer which are more or less injurious, according to the nature of the material used, but also serves to cover up and hide the results of unskillful brewing or unfit materials; giving to the public for consumption a liquor, that, if left to itself under natural conditions, would have become offensive to the senses and putrid with corruption long before it was offered for sale.

The only means of preservation allowed by the authorities in Germany and France is the process called, from the name of its author, "Pasteurization." This process is entirely rational and commendable,

¹ See Appendix A.

as it conduces to the preservation of the beer by destroying the germs of unhealthy ferments, not by simply paralyzing their activity as antiseptics do, and moreover it introduces no foreign constituents into the beer. Liquid carbonic acid is also coming into use in some of the larger Continental breweries.

Other preservative agents extensively employed at the present day are salicylic acid, bisulphite of lime, and boracic acid.

SALICYLIC ACID.

Salicylic acid ($C_7H_6O_3$) was first prepared by Piria and Ettling by oxidizing salicyl aldehyd, which had previously been obtained from various vegetable sources. It was afterwards obtained from oil of wintergreen, which is nearly pure methyl salicylate, a constituent also of many other essential oils. Its artificial production from phenol (carbolic acid) was discovered by Kolbe and Lautermann in 1860 but was not put into practical use until 1874, when Professor Kolbe succeeded in producing it at a moderate cost. It is now prepared almost exclusively in this way, the cheapness of the method having driven out of the market that which is prepared from oil of wintergreen.

By this process sodium carbolate is treated with dry carbonic acid gas and distilled at a rather high temperature, when one-half of the phenol combines with the sodium, forming sodium salicylate, while the other half is distilled over. The residue is decomposed with hydrochloric acid, the salicylic acid filtered off and washed, and purified by recrystallization or sublimation. The purest salicylic acid is obtained by dialysis, by which all the tarry matters can be separated. It is composed of long acicular crystals, having a peculiar, pungent, sweetish taste. It is irritating to the mucous membrane of the nasal passages and produces sneezing when handled.

The extended use of salicylic acid it owes to its property of arresting the action of ferments. This property has been extensively investigated and it is unnecessary to go into the subject further here.¹

In medicine, besides its use externally as an antiseptic, it is administered very extensively internally, its chief application being as a remedy for acute rheumatic fever. Its physiological action is given as follows in the United States Dispensatory, fifteenth edition, page 101:

When salicylic acid is given to man in doses just sufficient to manifest its presence, symptoms closely resembling those of cinchonism result. These are fullness of the head, with roaring and buzzing in the ears. After larger doses, to these symptoms are added distress in the head or positive headache, disturbances of hearing and vision (deafness, amblyopia, partial blindness), and excessive sweating. According to Reiss (*Berlinger Klin. Wochenschrift*, 1875, p. 674) decided fall of temperature, without alteration of the pulse, also occurs; but this is denied by other observers. The actions upon the system of the acid and of its sodium salts (also ammonium salt, Martenson, *Petersb. Med. Zeitschrift*, 1875, p. 343) appear to be identical, and, as sev-

¹ For recent information on this subject reference may be made to a paper by A. B. Griffiths, *Chemical News* .53, 28.

eral cases of poisoning with one or other of these agents have occurred, we are able to trace the toxic manifestations. Along with an intensification of the symptoms already mentioned there are ptosis, deafness, strabismus, mydriasis, disturbance of respiration, excessive restlessness passing into delirium, slow laboring pulse, olive-green urine, and involuntary evacuations. In some cases the temperature has remained about normal, but in others has approached that of collapse. The respiration seems to be characteristic, it being both quickened and deepened, often sighing. Sweating is usually very free, and the urine early becomes albuminous. Various local evidences of vaso-motor weakness may supervene, such as rapidly-appearing bed-sores at points subjected to pressure, and transitory dark colored macule on various parts of the body. In several cases death was probably produced by the acid, although there is scarcely one instance which is beyond doubt.¹ In certain cases the mental disturbance has been strangely prolonged, lasting for eight days. In some instances it is cheerful, in others melancholic in type. It is stated that upon drunkards the acid acts very unfavorably, violent delirium being an early symptom of its influence.

By the same authority the dose of salicylic acid to be employed in cases of acute rheumatism is given as one dram (3.9 grams) in twenty-four hours. It is excreted chiefly by the kidneys and may be detected in the urine very soon after its ingestion. Authorities in therapeutics warn practitioners of medicine against its administration to patients whose kidneys are known to be diseased, and of late years the opinion has been growing among physicians that it has a very irritating action upon these organs, many preferring the alkaline treatment of rheumatic fever on this account.

USE AS A PRESERVATIVE.

The "salicylic-acid question," as it is called, has received a great deal of attention for several years in Europe, and much has been written, pro and con, on the question of the propriety of its use as a preserving agent in articles of food and drink. In France its use as a preservative in any form of food or drink was forbidden by ministerial decree on the 7th of February, 1881. This decree was based upon a decision of the consulting committee of hygiene that its constant use was dangerous to health.

In Germany its use is prohibited, except in beers intended for export to other countries where its use is allowed.

Its prohibition in France called forth a great deal of opposition, and experiments were made and published, which were intended to show that its constant use in small doses exerted no injurious influence upon the system. Kolbe himself made experiments upon himself and his assistants by taking doses of .5 to 1.0 gram daily for several days, and found no appreciable ill effects to follow its use.² Whether such ex-

¹ In the case recorded in the *Virginia Medical Monthly*, June, 1877, forty-eight grains of the acid were taken in four hours. The symptoms were violent vomiting, headache, total unconsciousness, with stertorous breathing. Death occurred forty hours after the first dose.

² *Jour. prak. Chem.* 13, 106. Reference may be made to similar experiments, as follows: J. A. Barral, *Jour. de l'Agriculture*, 1882, 69. M. Blas, *Bull. de l'Acad. Royale de Méd. de Belgique*. Bd. 12, No. 9.

periments suffice to prove its harmlessness when used for many years and without regard to age, sex, or personal idiosyncrasy is still an open question. A most interesting and exhaustive discussion of the reasons for and against its use can be found in the report of the fourth meeting of the "Independent Union of the Bavarian Representatives of Applied Chemistry, at Nürnberg, 7th and 8th August, 1885,"¹ when this body refused, with but one dissenting voice, to grant its sanction to the proposed use of salicylic acid in beer in the quantity of .05 grams to the liter. Certainly no one would deny the advisability of at least restricting the amount to be used of so powerful an agent. In an article of daily consumption, and in consideration of the prevalence of kidney disease² at the present day, it is a matter worthy of grave consideration, whether it would not be more prudent to forbid its use altogether. At all events, beer in which it is used should be sold under its proper designation as "salicylated beer." It would certainly be of interest to the physician, who prescribes beer as a tonic to a weak convalescent invalid, to know if he were giving at the same time not inconsiderable doses of a strong therapeutic agent, expressly contra-indicated perhaps, in the case he has on hand.

The following amounts of salicylic acid were found in various articles of diet by Ch. Girard, director of the Municipal Laboratory, in 1881³:

Wine contained in the liter, 1.95, 1.60, 1.48, 1.41, 1.35, 0.81, and in one case even 3.50 grams salicylic acid.

Sirup contained in the liter, 0.5 to 1.50 grams.

Beer contained in the liter, 0.25 to 1.25 grams.

Milk contained in the liter, 0.25 to 1.85 grams.

It will be noticed that in one case of wine mentioned it contained in one liter the full therapeutic dose for twenty-four hours.

In this country but little attention seems to have been given to the use of salicylic acid as a preservative. In the investigation made by the New York State Board mentioned above, no search was made for it, or, in fact, for any other preservative. In the last year the municipal boards of New York and Brooklyn seem to have been taking cognizance of its extensive use, as is shown by the following extract from a paper read by Dr. Cyrus Edson, of the New York Board of Health, before the New York Society of Medical Jurisprudence and State Medicine, November 12, 1886:⁴

Within the past few months I have been confronted with a subject the importance of which to the community is very great. I have already touched upon it. It is the use of salicylic acid, a food preservative. Many, if not all, the manufacturers of preserved foods are adding small amounts of this substance to their goods to prevent loss by decomposition. The amount used is probably between one-third of a grain and a grain to the pound, and in the case of wines and beer, to which it is also added

¹ Published by Drs. A. Hilger and R. Kayser, Berlin, 1886.

² The most common form is popularly known as "Bright's disease."

³ Pharm. Cent. 22, 296.

⁴ American Analyst 1887, p. 7.

to prevent over-fermentation, from a grain to 3 grains to the pint in quantity. The French authorities, as I have said, believe that the use of salicylic and boric acids tends to irritate delicate digestive organs, and to also irritate the kidneys, through which they are eliminated under their own forms. Though I have talked with a number of scientific gentlemen in this country, few are willing to go into court and swear that this is also their opinion. It must not be lost sight of, however, that a person might at a meal take several articles of diet, each containing that which if taken alone would be a harmless dose, but taken together, and possibly for a considerable time, would prove highly injurious. The only safe way is to discard all additions to food which may possibly become a source of damage.

The following is of interest as establishing a precedent for the condemnation of articles containing salicylic acid:¹

Dr. Cyrus Edson, of the Board of Health, condemned and seized on November 11, 5,280 gallons of artificial wine in the possession of a Front street merchant. Dr. Edson reported the following as the process of manufacturing the stuff: "Dried fruits, such as raisins, currants, and peaches, are macerated with water, to which a certain amount of sugar is added. The mixture is then fermented, and when fermentation is considered sufficiently advanced it is checked by the addition of salicylic acid, sufficient being added to act as a preservative and prevent further fermentation. The so-called wine is then clarified, flavored, and colored to cause it to resemble port, claret, or any desired kind of wine. The object of the sophistication is to imitate and undersell natural native wines. The use of salicylic acid as a preservative is forbidden in France, as the French authorities consider it detrimental to health. I have consulted a number of noted chemists in this city as to their opinion concerning its use and nearly all unhesitatingly condemn it, holding that depressing effects in the nervous system would follow the daily use of the acid in small doses. It is my opinion and the opinion of Drs. J. B. Isham and J. B. Linehan, whom I have called on to assist in condemning and seizing the so-called wine, that the adulteration is a dangerous one and likely to cause sickness. The amount of acid used is about four and a half grains to the pint."

Recent information in regard to the status of the question in France, together with a very strong argument in favor of prohibiting entirely the use of this preserving agent, may be found in the following recommendation by Dr. Bartley:²

Dr. Bartley, the chief chemist of the Brooklyn Board of Health, has sent a communication to Health Commissioner Otterson, of that city, on the subject of poisonous beer adulterations, and strongly recommending that the Board take action against it. Dr. Bartley says: "During the year 1835 I had the honor to call the attention of the Department to certain abuses in the manufacture of lager beer, so called, in the course of which I said that 'the most important adulterations discovered, from a sanitary point of view, were yeast and sodium bicarbonate.' Since that time I have from time to time inspected the breweries of the city. As this beverage has become so largely used by families, it is now kept in bottles by many grocers. All brewers are compelled to manufacture an article for the use of bottlers. This is prepared with more care, in most cases, on account of the longer time it is to be kept before using. Unless it be thoroughly cured and well cleared the beer will often spoil before it is consumed, by a process of fermentation or putrefaction. To avoid the necessary care in the manufacture and the keeping of the beer for a longer time, it has become a practice among brewers to add salicylic acid to prevent this fermenta-

¹ American Analyst 1836, p. 416.

² American Analyst 1887, April 1.

tation after the beer is sent out. I have for some time been aware of this use of salicylic acid, as well as its addition to wines, canned fruits, cider, milk, and other goods, for the purpose of preserving them from fermentation. In fact, the use of this acid is coming into such general use in foods that it is becoming an important sanitary question as to its effects upon health when used in small quantities for a long time. There can be no doubt that in large quantities it acts very injuriously both upon the digestive processes and the kidneys. In its elimination the kidneys not rarely become acutely congested or even inflamed, giving rise to acute Bright's disease. Although a potent remedy in the treatment of acute rheumatism, it is not suitable for long administration, owing to the above injurious action. It requires the addition of from eight to ten grains of free salicylic acid to one gallon of beer in order to prevent the growth of ferments. If bicarbonate of soda has also been added, the quantity of acid necessary to be added is much more. Three grains have recently been found in each pint of wine. Assuming that the smallest effective proportion of the acid is used, viz, ten grains to the gallon, there are many persons in this city who take no inconsiderable amount of this drug every day of their adult lives. The salicylic acid of the market is prepared from carbolic acid, and is frequently contaminated with a small proportion of this very poisonous agent. This is a well known fact to all pharmacists. The sanitary question, then, rests upon the question of the action of small and long continued doses of salicylic acid, possibly contaminated with carbolic acid."

"Upon this point I think it necessary to quote here but one of many authorities. In 1881, and again in 1883, upon the recommendation of the Central Committee of Hygiene, the French Government prohibited the sale of articles of food containing salicylic acid. As protests were made against this legislation, and as these protests had led to contradictory judicial decisions, the above committee requested the opinion of the French Academy of Medicine. A commission created by that body of savants has recently made a report (*Bulletin de l'Acad. de Med.*, Paris, 1886, T. XVI., pp. 583 et seq.), from which the following is an extract: 'While in persons of good health the prolonged use of such small quantities of salicylic acid as would be contained in articles of food or drink treated with this substance is probably not injurious to health, it may nevertheless produce very decided disorders of health in certain persons, and especially in the aged and in those who have a tendency to diseased kidneys or dyspepsia. Salicylic acid and its salts are eliminated by the kidneys. They tend somewhat to check the action of the digestive ferments contained in the saliva, enteric juice, and pancreatic fluid, and hence to delay digestion; hence it is easy to understand that they may aggravate digestive or renal troubles.'"

"The report closes with the recommendation that the addition of salicylic acid or its compounds, even in small amounts, to articles of food or drink shall be absolutely prohibited by law. This commission found by their investigation that the quantities added to wine were about six grains to the gallon, and to beer from twelve to fifteen grains."

"From the facts here stated I am of the opinion that it is time that the addition of salicylic acid to articles of food received a check at the hands of sanitary authorities. I have made examinations of several different kinds of bottled beers manufactured and sold in this city, and have found a number of them to contain salicylic acid. The list examined contained some of the Western beers, which were also found to contain it. I would respectfully recommend that some action be taken by this Department towards the prohibition of this injurious adulteration.

"Respectfully,

"E. H. BARTLEY, M. D., *Chief Chemist.*"

SALICYLIC ACID IN SAMPLES EXAMINED BY THIS DIVISION.

Out of thirty-two samples analyzed by this Division I found seven to contain salicylic acid in sufficient quantities to admit of qualitative proof, or nearly one fourth of the entire number analyzed. The serial numbers of these beers corresponding to those in the large table on page 282 are as follows: 4801-3-5-6-17-23-25. These were all bottled beers, one being an imported (Kaiser) beer. None was found in any of the draft beers. Of the nineteen samples of American bottled beers, six contained salicylic acid, or nearly one third. These included the product of some of the largest breweries in the country, beers that are used to a very large extent all over the United States. Whether the acid is added in the breweries where the beer is made, or whether it is used by the local bottlers, I am unable to decide. In one case I found it in the beer sold here under the brand of a large Western brewery, and sent direct to the same brewery for another sample, which gave no test for the acid; unfortunately I cannot be sure in this case that the firm in question did not know the purpose for which the sample was intended.

DETECTION AND ESTIMATION OF SALICYLIC ACID.

Fortunately we have a particularly delicate and characteristic test for this substance, by means of which its presence can be detected in the minute quantity of 1 part to 100,000. This is the well-known characteristic violet color it gives with ferric salts. The test can sometimes be applied directly in the case of very clear beers, but in most samples it is obscured, either by the original color of the solution, or by the color produced by the combination of the iron with other constituents of the liquid. There are various procedures given for the separation of the salicylic acid from these constituents, so as to apply the test to it when in a pure state. Blas¹ has investigated various methods, including the previous precipitation of the beer with lead acetate, and the removal of the excess of lead with sulphuric acid;² shaking out the acidified solution with ether, evaporation of the ether and testing of the residue; treatment with bone-black, washing out the latter with alcohol and applying the test to the alcoholic solution;—none of which methods, he thinks, gives so good results as the application of the test to the urine of a person who has drank some of the beer in question. By this peculiar method of making the human body a medium of separation, he claims to make the test five times as delicate as where it is applied directly to the beer. Bornträger³ arrived at similar conclusions, and I can testify myself to its superior delicacy to the direct application to the beer. Muter⁴ was probably the first to use dialysis for the separation, also recommended by Aubry.⁵ Portele⁶ precipitates the tannin, &c., by a solution of gelatine and shakes up the filtrate with ether. Weigert⁷ uses

¹ Jour. prak. Chem., 19, 43.² E. Robinet, *Compt., rend.* 84, 1321.³ *Zeit. Anal. Chem.* 1881, 87.⁴ *The Analyst* 1, 193.⁵ *Zeit. Anal. Chem.* 1880, 105.⁶ *Weinlanbe* 1879, 389.⁷ *Zeit. Anal. Chem.* 1880, 45.

amyl alcohol as a solvent, as follows: "50cc. wine¹ (or beer) are shaken up with 5cc. amyl alcohol in a small flask for a few minutes and allowed to separate. The clear amyl alcohol is then drawn off and an equal quantity of alcohol added, with which it makes a colorless solution. To this solution is added a few drops of dilute solution of chloride of iron, which produces the violet color."

The use of chloroform as a solvent is recommended by the German Imperial Commission for the establishment of methods for wine analysis.²

The Paris Municipal Laboratory allows the choice of four different methods for applying the test in case the ether extract does not afford satisfactory results:

- (1) By adding a small quantity of a dilute solution of chloride of iron to precipitate the tannin, and subsequent extraction with ether.
- (2) By precipitation of the tannin with gelatine or albumen.
- (3) By treatment with ether in the ordinary way and after the ether is evaporated the residue is again treated with a few cubic centimeters of perfectly pure benzine, the solution evaporated, and the residue from this evaporation subjected to the test.
- (4) By treatment with pure chloroform.

The union of Bavarian chemists has³ adopted the method of Röse⁴ who uses a mixture of equal parts of ethylic and petroleum ether for extracting the beer or wine, as follows: "50cc. of the beer are shaken up in a separatory funnel with equal parts of ether and petroleum ether, after acidulating with 5cc. of dilute sulphuric acid. The separation follows very quickly, when the watery part is allowed to flow through the stop cock and the ethereal is poured out through the neck into a small dish. After the ether has been evaporated, and also the greater part of the petroleum ether except a few cubic centimeters, 3-4 cc. of water are brought into the still warm dish. This is well stirred, a few drops of a very dilute solution of chloride of iron added, and the whole filtered through a moistened filter, which allows of the passage only of the watery part of the solution. On the addition of the chloride of iron the petroleum ether solution assumes a deep yellow color, due to certain compounds of the iron with the resin of the hops. In the absence of salicylic acid the filtrate is nearly water-clear with a slight tinge of yellow; if present, even in traces, the solution takes on the well known violet color."⁵

To test the efficiency of these various methods two samples of a very dark beer known to be free from salicylic acid were taken, and to one was added salicylic acid in the proportion of .05 grams to the liter, and

¹ Some of the processes given are intended for wine, but are equally applicable to beer, milk, fruit juices, &c.

² Die Weinanalyse, Kommentar, u. s. w.

³ Op. cit.

⁴ Arch. f. Hygien. Analyst, 1886, 133.

⁵ The same test is given by H. Taffe, Bull. de la Soc. de Chim. de Paris, 46, No. 12.

to the other .005 grams. These two samples were then subjected to treatment by eight different methods, as follows :

- (1) Extracted with chloroform.
- (2) Extracted with ether simply.
- (3) Extracted with amyl alcohol.
- (4) Extracted with ether after previous precipitation with lead subacetate.
- (5) Extracted with ether after previous precipitation with gelatine.
- (6) Extracted with ether after previous precipitation with ferric chloride.
- (7) Extracted with equal parts of ethylic and petroleum ether.
- (8) Extracted with ether, the solution allowed to evaporate spontaneously, and the residue extracted with benzine.

These tests, tried on the beer containing .05 grams to the liter, gave results which ranged them in the following order of succession, according to the strength and brilliancy of the test: 8, 5, 7, 6, 2, 1, 4, 3. In methods 5 and 6 the length of time required to filter the solutions constitutes an objection to their use. The same tests applied to the beer containing .005 grams to the liter gave the test only in the case of Nos. 8 and 7, the former being the better of the two. The same series of tests were applied to a red wine, with very similar results, so I have adopted method No. 8 in all the work done on beers and wines, and would recommend either that or No. 7, both of which give a perfectly bright, water-clear solution, in which the slightest tinge of violet color is plainly visible.

QUANTITATIVE ESTIMATION.

While the qualitative determination of salicylic acid is so delicate and easy of execution, the quantitative estimation is unfortunately a tedious and unsatisfactory operation, in the small quantities in which it is found. This is an argument in favor of the entire prohibition of its use in foods in preference to the restriction of the quantity to be used.

The determination may be made by the same method as described above for the qualitative test, simply making the extraction complete. One hundred cubic centimeters of the beer or wine are taken, acidified with a few drops of hydrochloric acid, and extracted with three successive portions of ether of 50cc. each; these are mixed and the whole allowed to evaporate spontaneously. The residue from this evaporation is heated for an hour on the water bath to drive off volatile acids, and treated with 150cc. of pure benzine, which is allowed to stand in contact with the residue for twenty-four hours, when it is drawn off carefully and the residue again treated with 50cc. of benzine, which is added to the first portion. This 200cc. of benzine is then made up to 500cc. with absolute alcohol, and titrated directly with a decinormal solution of soda, previously standardized by operating upon a similar mixture.

The acid may also be extracted by treatment with successive portions of chloroform, which is carefully decanted, the solution evaporated, and the crystallized salicylic acid weighed.¹

A. Rémont² published in 1881 a method for the colorimetric estimation of salicylic acid, which was modified by Pellet and De Grobert³ in the following manner:

A series of uniform test-tubes are prepared about 20cm. in height and 15mm. in caliber, to which are added, successively, 1cc., 0.75cc., 0.5cc., 0.4cc., 0.3cc., 0.2cc., 0.1cc. of a solution of 1 gram of salicylic acid in 1 liter of distilled water, and the volume in each tube brought to 10cc. with distilled water. To the first tube is added three drops of a dilute solution of ferric chloride (1005 to 1010 specific gravity), in the second and third two drops, in the others one drop. The last tube may be simply stirred with a glass rod which has been dipped into the iron solution. One hundred cubic centimeters of the beer or wine to be tested are now taken and shaken up with 100cc. of ether, and five drops H_2SO_4 of 30° B., allowed to stand, carefully separated, and this operation repeated twice. The ether is quickly evaporated off in the water bath, the residue brought into a porcelain evaporating dish of about 6–8 cm. diameter, the flask washed out with a few cc. of ether and the dish placed in an air bath heated to about 50° C. to drive off the ether completely; 1.5cc. of a solution of soda is then added, of such strength that 10cc. contain 0.4 grams Na_2O , which is sufficient to saturate about 0.2 grams of salicylic acid, equal to a content in the sample of 2 grams per liter. If the residue is still acid after this addition, it is due to acetic acid. By evaporating to dryness this excess can be driven off while the salicylic acid is retained, as it is able to displace acetic acid from its salts. The residue is now treated with five drops of H_2SO_4 of 30° B., and then with 20cc. of benzine, and the whole filtered; 10cc. of the filtered benzine solution is brought into a test-tube of similar dimensions to those mentioned above; 10cc. distilled water and one or two drops of the dilute ferric chloride solution added, and the contents well shaken. If salicylic acid is present it is all taken up by the lower watery portion and the color may be compared with that of the standard tubes. If it agrees in intensity with one of these, the calculation is very simple. Suppose, for example, it agrees with the fourth tube, which contains in the 10cc. of liquid 0.0004 grams salicylic acid, then the 10cc. of benzine solution also contained 0.0004 grams, and the 20cc. from which it was taken contained 0.0008 grams in 100cc. of the wine, or .008 grams to the liter. The authors of this method found on applying it to wine to which a known quantity of salicylic acid had been added, that only 93 per cent. of the amount added was found, so their results were divided by 93 on that basis. With this modification they obtained from two samples of

¹ These two methods are employed in the Municipal Laboratory of Paris.

² Jour. Pharm. Chim. [5], 4, 34, Chem. Cent., 1881, 773.

³ Compt. Rend. 93, 278, Chem. Cent., 1881, 711.

wine to which had been added, respectively, 0.084 and 0.126 grams per liter 0.084 and 0.123 grams.

M. Rémont¹ also devised, in 1832, what might be called an empirical method for the estimation of the quantity of salicylic acid added to wine or beer, which would doubtless prove very convenient in case its use were limited by law to a certain definite quantity, as follows:

In a liquid, similar to that which is to be tested, is dissolved a known quantity of pure salicylic acid; of this standard liquid 50cc. are taken and well shaken with 50cc. of ether, and allowed to separate; 25cc. of the ether are taken in a flat dish and subjected to evaporation at a temperature below boiling, in the presence of 10cc. of water; when the ether has disappeared, the water is poured into a graduated cylinder and its volume made up to 25cc. with the washings of the dish. This solution contains the same proportion of salicylic acid as the standard; 10cc. of the liquid to be analyzed are then taken and shaken with 10cc. of ether; 5cc. of the clear ether are taken, evaporated with 2cc. of water, and the residual liquid made up to 5cc. with the washings of the dish, as above.

Two tubes, each graduated to 30cc., are taken, and into one is introduced 5cc. of the standard aqueous solution as obtained above, and into the other the 5cc. obtained from the sample for analysis. To each tube is added the same quantity of a 1 per cent. solution of ferric chloride, avoiding an excess. The comparison of the two tubes may then be made, and the process can be made quantitative by diluting with water, or other colorimetric methods.

The author insists upon the necessity of taking, as a means of comparison, a liquid of the same nature as the sample for analysis, as the foreign matters which ether dissolves from wine, beer, or cider alter the delicacy of the color test.

SULPHITES.

The use of sulphurous acid as a preservative agent in beer and wine, either in the form of soluble sulphites, liquid sulphite of lime, or sulphur fumes, is not at all recent. It is one of the oldest preservatives known. Together with other chemical preservatives its use is forbidden in France, and the German authorities include it with borax as an agent whose physiological effect is still too little known to allow of its indiscriminate use. It is also sometimes introduced into beers by the hops, which are very generally preserved by means of sulphur fumes. The Bavarian authorities allow its use in sulphuring barrels and hops, as will be seen when their method of analysis is described later. Of course the quantities brought into the beer in this way are very small.

The qualitative test, which is given by many of the books on the subject,² viz, the reduction of the sulphur to hydric sulphide gas by means of nascent hydrogen, is entirely erroneous, as I have proved by experi-

¹ Compt. Rend. 95, 786.

² König, for instance, p. 410; Dietzsch, p. 123.

ments made upon the various albuminous constituents of beer with the same test. Hops (known to be free from SO_2), malt, and even ground barley, treated with hydrochloric acid and zinc gave a very distinct blackening of lead acetate paper in the course of fifteen minutes, and the test applied to the beers examined gave a distinct reaction *in every case*. I concluded from the above-described experiments that the H_2S came from the sulphur contained in the albuminous bodies of the grain, which was reduced by the nascent hydrogen. Blank experiments with the reagents used gave no test for sulphur. Since I made these experiments similar conclusions were reached by M. von Klobulow,¹ who found that sulphur was reduced from any of its compounds by nascent hydrogen, and so complete is this action that he has made it the basis for a new method of estimating sulphur. It was probably by this test that sulphurous acid was found in very old wines, as has been reported.

The method of detecting the presence of SO_2 by its oxidation to H_2SO_4 is probably the best, and can be employed very successfully for the quantitative estimation also. There are various methods in use for affecting the oxidation, as well as for the subsequent determination of the sulphuric acid formed. In the method employed by the Paris Municipal Laboratory the beer is acidulated with sulphuric acid, and a current of pure carbonic acid gas is drawn through the liquid and then into a solution of chloride of barium mixed with iodine water. If SO_2 is present, a precipitate of sulphate of barium forms in the latter mixture. Other oxidizing agents may be used instead of the iodine. Wartha² used nitrate of silver solution for a qualitative test; bichromate of potash furnishes a very convenient agent, and the solution of it may be made standard and titrated afterwards to determine the extent of oxidation. I have used permanganate of potash with very good results for a qualitative test. But probably the best method for both qualitative and quantitative determination is that used by the union of Bavarian chemists, which I have employed in testing the samples examined. It is as follows: 100cc. of the liquid to be examined are acidulated with phosphoric acid, and distilled in a stream of carbonic acid gas, and the distillate received in a flask containing 5cc. of normal iodine solution. After the first third is distilled off, the distillate, which should still contain excess of free iodine, is acidified with hydrochloric acid, heated, and barium chloride solution added. If a precipitate of more than 10 milligrams is obtained in the barium solution, the wine or beer contains sulphurous acid in excess of the legalized limit. (The allowance of 10 milligrams of barium sulphate is made to admit of the hops being sulphured.) In using this method I have found it necessary not only to have the delivery tube from the condenser dip into the iodine solution, but also to attach a mercury valve to the flask in which it is received.

¹ Zeit. Anal. Chem. 25, 155; Chem. News, 1886, 325.

² Berichte d. Deutsch. Chem. Gesell. 13, 657.

A few of the samples examined by this test gave a slight turbidity with barium chloride, viz: Serial Nos. 4804-6-10-13 and 14, while only one, No. 4815, gave sufficient precipitate to justify the assertion that a sulphite had been added to it. I have not been able to find any recorded instance of sulphurous acid being found in American beers.

BORAX.

This agent, although used very extensively in preserving meats, vegetables, and canned goods, does not seem to have been applied to malt liquors to any great extent, although it has been found in wines. Its use is prohibited in France and Germany. The test for boracic acid is best applied to the ash. If this is rubbed up with water acidulated with a little hydrochloric acid and a piece of turmeric paper dipped into the solution and then dried, it will show a peculiar reddish tint if borax be present. For a very delicate test a large quantity of the liquid to be tested may be evaporated to a sirup, with a slight addition of sulphuric acid, the residue extracted with alcohol, and the latter ignited. The edges of the flame will be colored green if borax is present. None of the samples examined gave any test for borax.

In conclusion of the work on preservatives, it may be noted that it was done during the cold weather of January, February, and March. It is quite probable that during warm weather the use of preservative agents is still more general than shown by the analyses.

MINERAL ADDITIONS.

The presence of lead, copper, or zinc, sometimes observed in malt liquors, is due usually to the use of brass faucets or lead pipes by the retailer in drawing off the liquor or in filling bottles. The amount of these metals taken up by acid liquors in this way is quite small usually, but may be considerable if they are long left in contact with the metallic surface. Thus the first glass drawn from a faucet in the morning is apt to contain considerable copper and zinc in solution. In Paris the apparatus used for drawing beer is subject to supervision, and a frequent cleansing and proper kind of material is insisted on. The Brooklyn Department of Health issued an order in 1886 prohibiting the use of unprotected brass faucets in drawing beer, but its enforcement has not been insisted on.¹ Analyses made for the board by Otto Grothe of ales drawn through pumps showed small quantities of copper, zinc, and lead in every case.²

Alum is sometimes used as a clarifying agent in the brewing of beer. The method of detecting the presence of the metals in liquors need not be dwelt on here. It is best performed in the ash from a large quantity of the suspected sample.

¹Annual Report Dept. Health, City of Brooklyn, 1886, p. 87; and 1887, p. 63.

²Ibid.

BICARBONATE OF SODA.

This salt is added to beer for the purpose either of correcting an undue acidity of the beer, resulting from improper brewing, or of imparting to it an increased "head," or content of carbonic-acid gas, or for both purposes. The salt is decomposed by the free acid of the beer and the gas liberated, lactate and acetate of soda being left dissolved in the beer. This seems to be purely an American practice; at least I have failed to find any mention of it in European authorities. Some of them mention the use of marble dust or magnesia for the correction of acidity, but very little consideration is given to the subject. In this country, however, it seems to be very widespread. The following extracts are taken from a paper read by Otto Grothe, Ph. D., before the American Society of Public Analysts:¹

The Health Department of the city of Brooklyn has for some time carried on investigations with reference to the brewing of lager beer as practiced in that city. The peculiar cathartic effect of some of the Brooklyn beers seemed to indicate the presence of some substitutes, principally for hops. The analysis of such suspicious beer failed, however, to reveal anything of importance, either on account of the absence of such substitutes or because the quantities of beer used were too small. Before going to the expense of purchasing a keg or two from each brewery for the chemical laboratory, Dr. Bartley thought it to be the best to have the breweries, in the first place, inspected. These inspections resulted principally in the discovery of a variety of substances used by beer brewers as clearing and improving agents, the latter being considered the most objectionable. They were sodium bicarbonate, tartaric acid, cream of tartar, isinglass, or gelatine, glucose, grape sugar, juniper berries, and salicylic acid.

Sodium bicarbonate is a substance more regularly used by brewers. * * * The opinion of the brewers about the necessity of this addition is very much divided; while some believe it to be utterly necessary, others say they would rather do without it, as it causes them a heavy expense. They all say, however, they cannot avoid it, because the public wants a perfectly neutral beverage. There is beer in the market which has no addition of bicarbonate. * * * The quantity of soda added varies very much, and we may say in proportion to the quantity of acid contained in the beer. This quantity of acid in the beer depends upon the knowledge and the attention of the brewer. Thus we find that breweries which have clean, well-ventilated, and flushed cellars, in which refrigerating machines are in use, and which are conducted in a scientific way by an expert foreman, can afford to sell their product with less than one-third of the quantity of sodic bicarbonate used by smaller concerns which are not so well conducted and which have not the facilities of their larger competitors.

The largest quantity of bicarbonate used is about $2\frac{1}{2}$ ounces to the keg, or quarter of a barrel. The size of a barrel varies from $31\frac{1}{2}$ to 33 gallons, according to the age, the older kegs becoming smaller by the contraction of the wood. A keg, therefore, contains 8 gallons of beer, or 64 pints, which is considered equal to about a hundred glasses as sold in the beer saloons over the counter. A glass of beer, therefore, contains in some cases three-fourths of a gram of bicarbonate of soda; and as a moderate beer drinker will, under certain circumstances, for instance in hot weather, drink about twenty glasses of beer a day, he takes about 15 grams, or 252 grains of bicarbonate of soda with it. A heavy beer drinker—say, a laborer who works outdoors and who buys the beer by the pint—may consume as many as forty glasses a day, and he takes an ounce of bicarbonate of soda with it. The smallest quantity of bicarbonate of

¹ Ann. Rep. Dept. of Health, City of Brooklyn, 1885, p. 92.

soda used in our breweries is 1 ounce to a half barrel, and the difference in the effect of that addition is a very remarkable one, the beer tasting slightly acid.

There cannot be any doubt that large quantities of bicarbonate of soda regularly introduced into the stomach are detrimental to the health. Inasmuch as the lager beer is used as a food by many people, it would be greatly appreciated by intelligent beer-brewers and beer-drinkers if the use of bicarbonate of soda could be regulated by the authorities, or, if possible, entirely abolished. By such regulations the unclean brewer would be compelled to either keep his brewery clean, or go out of the business altogether. Such regulations should also be extended to the quality of the metals of the apparatus used in the different brewing processes, so that to the American lager beer the same name can be given as to the German beer, which Justus von Liebig called "liquid bread."

There are several rather misleading statements in the above. Dr. Grothe says in the first place that "the public wants a perfectly neutral beverage," which is open to considerable doubt; and again, "the smallest quantity of bicarbonate of soda used is one ounce to a half of a barrel, and the difference in the effects of that addition is a very remarkable one, the beer tasting *slightly acid*." If this latter statement is taken in a strictly chemical sense, it is rather paradoxical, for a bicarbonate added to a liquid of course tends to make it alkaline. What is meant by its tasting slightly acid doubtless is that it acquired a pungency to the taste on account of the liberation of carbonic acid gas from the bicarbonate by the free acid existing in the beer. One of the beers I examined (No. 4816) was actually alkaline in reaction from excess of added bicarbonate, and the taste was far from being agreeable.

I would hardly take so decided a stand as Dr. Grothe in regard to the injury done to the health of the beer-drinker by bicarbonate of soda *per se*. It may be necessary to explain to a non-scientific reader that the bicarbonate does not remain in the beer *as bicarbonate*, unless there is an amount added in excess of the quantity of free acid present in the beer. This free acid (mostly acetic in soured beers, but due chiefly to acid phosphates in normal beers) combines with the bicarbonate, setting free carbonic acid, and forming acetate of soda and basic phosphate, which remain in solution. The reaction is very similar to that which takes place in using baking powders for cooking purposes, except that in the latter case tartrate of soda and potash (Rochelle salts) is left instead of acetate and phosphate of soda. Where bitartrate of potash is added to the beer along with the soda (as sometimes occurs according to the Brooklyn report) the reaction is precisely the same. In these days of the almost universal consumption of baking powders there is doubtless enough alkaline salts thrown into a man's stomach with his food without pumping them in with his drinks as well. At all events there can be but little question of the propriety of prohibiting the use of bicarbonate of soda in beer. It is entirely unnecessary and foreign to the production or preservation of pure beer. Moreover, its use serves to cover up and hide the effects of poor brewing and improper storing or refrigerating, and should be prohibited from this cause alone if there were no other.

DETECTION.

The detection of the addition of very small quantities of bicarbonate of soda to beer is by no means an easy matter when the constant presence of soda salts in beer ash is considered, and the very variable content of alkali in the waters used for brewing purposes. The ash of beer is of very variable composition, being obtained in part from each of the principal constituents which enter into the preparation of the drink, viz, the malt, the hops, and the water used in the brewing. The content of soda (Na_2O) in the ash varies in different published analyses, from less than 4 per cent. up to 35 per cent. of the ash,¹ and this without a sufficient proportion of chlorine to account for the large per cent. of soda as salt. The presence of any considerable quantities of carbonate in beer ash, however, is abnormal, and indicates the addition of bicarbonate of soda to the beer, the acetates, lactates, &c., formed from it being converted into carbonates by the process of incineration. So far as I have been able to ascertain, no carbonic acid has ever been found in the ash of normal beer, its alkalinity being due to the presence of alkaline phosphates. I have found no statement as to whether the ash of normal beer reacts acid or alkaline in any of the books on the subject except Elsner,² who says it reacts acid. This must certainly be a mistake, for the ash of every sample I examined gave a strong alkaline reaction, requiring from 5 to 3.5cc. of decinormal acid for the neutralization of the ash from 100cc. of beer. Hassall³ says, on the other hand, "the alkalinity of the ash must be estimated" in determining whether some alkaline earth or alkali has been added.

In order to investigate this question I procured a sample of beer made in Lafayette, Ind., which was guaranteed to contain no bicarbonate of soda, and which, from my knowledge of the parties through whom it was obtained, I have every reason to believe to be a sample of perfectly pure beer.⁴

The complete analysis of this sample was as follows:

Specific gravity	1.0201
Per cent. alcohol by weight.....	3.72
Do. alcohol by volume.....	4.64
Do. extract	6.34
Do. reducing sugar as maltose	1.42
Do. ash286
Do. fixed acid as lactic201
Do. volatile acid as acetic049
Do. phosphoric acid059
Do. carbonic acid238
Normal polarization	+69.7

Several portions of 100cc. each were taken, various quantities of bicarbonate of soda added, and after solution each portion evaporated

¹ See Wolff, Aschen-Analysen, p. 23.

² Page 89.

³ Page 706.

⁴ A small quantity of rice grit was admitted to have been used in its manufacture.

to dryness and burned to ash. The alkalinity of the ash was then ascertained, with the following results:

	No. 1.	No. 2.	No. 3.	No. 4.
Amount, in grams, of bicarbonate added....	None1	.5	1.0
Number of cubic centimeters decinormal acid required for neutralization.....	1.5	5.	17.5	20.

From these results it will be seen that the alkalinity of the ash shows very plainly the addition of considerable quantities of bicarbonate, but small additions would not be detected in this way. Again, the books state that the addition of bicarbonate of soda can be recognized by the strong effervescence of the ash with an acid. This statement is based entirely upon theoretical grounds, which have been shown by later investigations to be fallacious. When the acidity of normal beer was supposed to be due chiefly to lactic acid, the formation of lactate of soda, which would be converted into a carbonate on ignition, would, theoretically, make an ash which would effervesce strongly with acid. But now that it is known that the acidity is due to acid phosphates, it is easily seen that the addition of bicarbonate would only tend to the production of neutral or alkaline phosphates, which would give no effervescence with acid. This is shown by experiments I made with the sample of normal beer. Several portions of 100cc. each were taken, and different quantities of bicarbonate of soda added, and when solution had been effected the beer evaporated, and the residue carefully incinerated. The test for the presence of carbonate was made very carefully, by pouring a few drops of water on the ash, and turning the whole into a test-tube containing dilute acid.

No.	Amount bicarbonate of soda added.	Remarks.
	<i>grams.</i>	
1.....	None	No effervescence.
2.....	.1	Do.
3.....	.2	Do.
4.....	.5	Slight effervescence.
5.....	1.0	Strong effervescence.

It will be seen from the above that there was no carbonate in the ash until sufficient bicarbonate was added to the beer to neutralize all its acidity, leaving an excess of bicarbonate in the beer. In Nos. 4 and 5 the beer reacted alkaline before evaporation.

The same beer was allowed to stand until it had become quite sour and spoiled, when the above set of experiments was repeated, with the following results :

No.	Amount bicarbonate added.	Remarks.
	<i>grams.</i>	
1.....	None	No effervescence.
2.....	.1	Slight effervescence.
3.....	.2	Strong effervescence.
4.....	.5	Do.
5.....	1.0	Do.

From the above results it would seem that the bicarbonate united by preference with the acetic and lactic acids formed rather than with the acid phosphates of the beer, though it would require more experiments on the subject to thoroughly establish this point. If such is the case, however, it will be possible to detect the addition of bicarbonate to a beer that has soured, though not to a normal beer. The smallest quantity used in practice, according to the Brooklyn report, is one ounce to a half a barrel, which would be about 1 to 2,000, while the addition of .1 gram to 100cc. would be 1 to 1,000. Girard¹ gives as a test for the addition of bicarbonate of soda to ciders the following procedure : Decolorize with bone-black, evaporate to dryness, treat the residue with alcohol, which dissolves the acetates, which can be detected in the alcoholic solution. I have tried this test with very indifferent results, as I find the alcohol dissolves so much other matter from the dried residue that it is difficult to detect the presence of acetic acid in small quantities. The distillation of the beer in a current of steam² after the addition of phosphoric acid offers a better method for the separation of acetic acid, which may be searched for in the distillate, and if found to be present in any quantity, while the acidity of the beer itself is normal or below normal, the addition of bicarbonate of soda will be pretty well established. In this way I established the fact of the addition of bicarbonate in Nos. 4814 and 4816, which were the only samples of which I could be positive; and although I strongly suspected several of the other samples, owing to the difficulties of the test I would not pronounce positively upon them.

SALT.

A variable quantity of chloride of sodium is a normal constituent of all beers, being derived principally from the water used in the brewing. Even a slight further addition of salt might be deemed admissible to properly "season" the beer to the taste, just as breadstuffs are treated. Many brewers, however, are in the habit of adding a large quantity, either for the purpose of covering up some objectionable taste, or of increasing the thirst of the consumer. The English Government places

¹ Report Paris Municipal Laboratory.

² See under "Free volatile acids in wines," page 342.

the limit of chloride of soda which might come from the normal constituents at 50 grains to the gallon, or about .086 per cent., and treats any excess of that amount as evidence of an improper addition. This standard is undoubtedly a very generous one. Dr. Englehart found quite a large number of the samples examined by him to overstep the limit of 50 grains to the gallon, one sample containing as high as .338 per cent. Of the samples examined here none were beyond it.

ESTIMATION.

The estimation is very readily carried out on the ash, either gravimetrically or by a standard solution of silver nitrate with potassic chromate as indicator. For careful work the ash should be simply charred, so as to avoid loss of the chloride by volatilization, and the charred mass extracted by repeated additions of small quantities of hot water.

CLOUDY BEER.

Cloudiness in beer is sometimes due to the separating out of albuminous matter from changes in temperature, but usually to the presence of yeast, the fermentation not having been complete. This condition of things is best detected by means of the microscope, which shows the presence of quantities of yeast cells, and, in case other fermentations have set in, of their characteristic bacteria. "Yeast-cloudy" (*hefe trübes*) beer is considered unhealthy in Germany, and it is considered one of the qualifications of a good beer that it shall be absolutely bright and clear. An extensive investigation of the unhealthfulness of yeast-cloudy beer lately made by Dr. N. P. Simonowsky¹ in Pettenkofer's laboratory, who found that such beer had a disturbing effect in both natural and artificial digestion, producing in persons using it obstinate catarrh of the stomach, which persisted for some time. Both Simonowsky and Pettenkofer conclude that the sale of yeast-cloudy beer should be prohibited.

The Bavarian chemists at their last meeting at Würzburg, in August, 1886, adopted the following resolution in relation to yeast-cloudy beer:

Beers which are incompletely fermented for use must be entirely free from yeast; that is, must not contain yeast in a cloudy suspension.

¹Zeit. für das gesammte Brauwesen?, 9 Jahrg. 1886, No. 7, 8, 9; abstract Bied. Cent., 1887, p. 70



PART II.

WINES.

WINES.

The statistics in regard to the consumption and production of wines can be observed by referring to the table given under malt liquors (page 267), where it will be seen that in the year 1886, 22,067,220 gallons were consumed, of which 17,366,393 gallons were produced in this country. The consumption per capita has not increased very greatly during the forty-six years since 1840, but the total amount consumed has increased very greatly, it being less than 5,000,000 gallons in 1840. It will be noticed also that the amount produced in this country in proportion to the amount imported has increased to a remarkable degree. In 1840 there was about thirty-eight times as much wine imported as was produced in this country; in 1886 the amount of domestic wine consumed was nearly four times as great as the amount of wine imported. This does not fully represent the production, however, for it does not include the exports, which have increased very greatly of late years, as I am reliably informed, although I have no accurate data upon this point. The largely increased domestic production is principally due to the development of the industry in California.

The following table shows the relative rank of this country among the wine-producing countries of the world; it is taken from the same source as the preceding statistics:

WINE PRODUCTION OF THE WORLD.

Average production of wine in the principal wine-growing countries of the world.

[Estimate by M. Tisserand in 1884, taken from "Journal of the Statistical Society," London, 1885.]

Countries.	Production.	Countries.	Production.
	<i>Imperial gallons.</i>		<i>Imperial gallons.</i>
France	765,175,972	Greece	28,600,000
Algeria	722,000,000	United States	18,000,000
Italy	605,000,000	Turkey	22,000,000
Spain	484,000,000	Cape of Good Hope	15,400,000
Austria-Hungary	187,000,000	Roumania	15,400,000
Portugal	88,000,000	Servia	11,000,000
Germany	81,290,000	Australia	1,933,800
Russia	77,000,000		
Cyprus	35,200,000	Total	2,485,599,772
Switzerland	28,600,000		

PREPARATION OF WINE.

The growing of grapes for wine and the proper treatment of the juice for its conversion into wine have formed the subject of numerous treatises, that branch of technology having received a great deal of attention and study in countries where it is carried on. Only a short sketch of the leading features of the process can be given here, necessary to a proper understanding of the product itself.

Wine is properly the pure fermented juice of grapes; its composition is very variable, and the differences in the varieties of grapes used admit of almost endless modifications of the product obtained from them. Moreover, many other conditions affect more or less the composition of wine, as the nature of the soil, the climate, the method of cultivation pursued, the weather during the particular season when the grapes were ripened, &c. Thus the same variety of grapes when grown under different conditions of soil, climate, &c., produces different wines, and even in the same country the same variety of grape produces wines varying considerably in different seasons.

The most important constituent in the grape is its sugar, from which the alcohol is formed, so as a general rule the grapes are allowed to become fully ripened before they are removed from the vine. The first step is the production of the *must*. To this end the grapes are first bruised and crushed, either by the aid of machinery or by the more primitive but very effective method of trampling them by the feet of men. In some cases, and for very fine wine, the woody stems are removed from the crushed grapes (*dérâpage*). In other cases, especially in white wines, they are left, their contents of tannin making them a desirable addition to the grapes. To obtain the juice the grapes are subjected to pressure. The amount obtained varies with the means employed, the kind of grape, &c., but may be stated at about 60 to 70 per cent. of the weight of the grapes. For red wines the juice is allowed to stand in contact with the skins a variable length of time until it has acquired from them the desired depth of color, and in this case the fermentation commences before the juice is expressed. All musts contain pretty much the same proximate principles, their differences being due solely to the relative proportions of the different constituents. Briefly stated, these constituents are as follows:

1. Saccharine matter (chiefly dextrose), which may constitute as high as 25 to 30 per cent. of the must.
2. Albuminoid matter.
3. Gummy matter, pectin, &c.
4. Extractive matter, illy-defined substances, comprising the coloring matters, if any, the flavoring matters, &c.
5. Organic acids and their salts, comprising malic acid (especially in bad seasons), a slight trace of tannic acid derived either from the stems or skins, and tartrates of potassium and calcium.
6. Mineral matters: Phosphoric, sulphuric, hydrochloric, and silicic acids combined with potassium, sodium, iron, and magnesium.

7. Water, 70 to 90 per cent.

The must is fermented in suitable vats of wood or stone, according to the usage of the country; the fermentation is produced spontaneously, that is, by germs accidentally introduced into it from the air or on the surface of the grapes themselves. If the fermentation does not take place promptly it is started up by introducing into it a supply of yeast-cells from some must which is already in a state of fermentation. Sometimes a small quantity of must is fermented in anticipation of the vintage season as a "sponge," its fermentation being first induced by a small quantity of well washed beer yeast. The use of albuminous yeasts, such as bread yeast, &c., is generally avoided as much as possible, however, as tending to produce lactic and acetic or other objectionable fermentations entirely incompatible with the production of a wine with a delicate flavor.

The temperature at which the fermentation is carried on has a very decided influence upon the character of its product, and the practice differs in different countries in this respect. In California, Spain, South of France, Austria, and Hungary fermentation is conducted at a comparatively high temperature, 15° to 20° C., while in Germany a low temperature, 5° to 15° C., is employed. As with beer, the yeast of either variety of fermentation, high or low, reproduces the same kind of fermentation in musts to which it is added, but the subject of the different ferments, as applied to wine, has not been so carefully studied as with beer. The high fermentation is said to give a wine rich in alcohol, but lacking in bouquet, while the reverse is the case with the low fermentation.

The duration of the fermentation varies with the temperature, the amount of sugar to be transformed, &c.; the completion of the process may be known by the cessation of the disengagement of carbonic acid gas and by the diminution of the specific gravity of the liquid, so that the areometer marks zero or less.

After fermentation is complete, the wine is drawn off from any sediment it may contain into casks or barrels, where a second slow fermentation takes place, continuing sometimes several months. When it is over, the wine is "racked off" into fresh casks, which are closely bunged up. The operation of racking off may have to be repeated several times, and it is sometimes necessary to add isinglass, or other gelatinous material, which serves to clarify the liquid, acting on the tannin which it contains. This operation is called "fining."

CHANGES PRODUCED BY FERMENTATION.

The principal change in the chemical constitution of the must produced by fermentation is the conversion of the sugar into alcohol and carbonic acid. One hundred parts of sugar produce 50 parts of alcohol, in round numbers. All the sugar, however, is not converted into alcohol and carbonic acid; a small part is converted into glycerine and succinic acid.

The bitartrate of potash, being insoluble in alcohol, is gradually deposited as the content of alcohol in the wine increases, and forms the substance known as "argol" or crude tartar. This distinctive constituent, tartaric acid, constitutes the superiority of grapes, over other fruits for wine-making purposes, the comparative insolubility of its acid salts furnishing a means of removing the excess without the addition of other chemical agents.

Other changes take place, especially during the slow second fermentation, not so well defined or so well understood as those mentioned, but of great importance in their relation to the quality of the final product. These changes, which continue after the fermentation has ended, constitute what is called the "ageing" of the wine and produce its "bouquet" or flavor, generally attributed to the etherification produced by a slow action of the acids upon the alcohols. Wine improves with age, but there is a limit after which it degenerates again and loses its flavor.

METHODS FOR "IMPROVING" WINES.

In France and Germany several methods are in use for increasing the yield of wine or improving its quality. These are especially resorted to in unfavorable seasons, when the want of sufficient sun prevents the formation of enough sugar in the grape and the proportion of acid is high.

Chaptalization consists in neutralizing the excess of acidity in the must by the addition of marble dust, and increasing the saccharine content by the addition of a certain quantity of cane sugar, which the vintners sometimes replace by starch sugar. In this process the quantity of the wine is not increased, but it becomes richer in alcohol, poorer in acid, and the bouquet is not injured. It is much used in Burgundy.

Gallization, which was invented by a German, Dr. Ludwig Gall, has for its object the production of a standard must, which shall contain a definite proportion of acid and sugar. This is brought about by the analysis of the must and the addition to it of water and sugar, the quantity to be added being ascertained by reference to tables.

Petiotization.—This process, which takes its name from Petiot, a proprietor in Burgundy, is carried out as follows: The *marc* from which the juice has been separated as usual by pressure is mixed with a solution of sugar and water, and the mixture again fermented—the second steeping containing, like the first, notable quantities of bitartrate of potash, tannic acid, &c., which are far from being exhausted by one extraction. The process may be repeated several times, the different infusions being mixed. This process is very largely used in France, and is said to produce wines rich in alcohol, of as good bouquet as the original wine, and of good keeping qualities. It is not allowed to be sold there, however, as *natural* wine.

To what extent these methods obtain in this country I am unable to state. It is probable, however, that they are but little used, as the principal fault found with American wines is their deficiency in bouquet,

not in their content of sugar. The detection of wines made in any of the above-mentioned ways is rather a difficult matter chemically, and requires a knowledge of the composition of the pure product only obtained from large numbers of analyses, extending over many years; which data, although existing in abundance in European countries, are, as yet, lacking here, owing to the comparatively recent development of the industry and the small amount of work done on the subject.

PRESERVATION OF WINE.

The method *par excellence* for the preservation of wines is Pasteurization, already alluded to in this report on malt liquors. The temperature employed is from 50° to 65° C., and serves to completely destroy all vegetable life in the wine. When a process so unobjectionable in every way answers its purpose so admirably, it furnishes an additional argument in favor of the legal suppression of all chemical means of arresting fermentation by the use of antiseptics, &c.

In regard to the use of antiseptics for the preservation of wines, I cannot do better than to give the opinion of Prof. E. W. Hilgard, of the University of California, who has probably done more than any other one man towards placing the wine industry of California upon a scientific basis, and whose work, published in the Bulletins of the State Agricultural Experiment Station, I shall have frequent occasion to refer to in the course of this investigation.¹

* * * * *

Addition of antiseptics.—As before stated, any of the fermentations above referred to may be stopped by the action of the substances known as disinfectants, antiseptics, or poisons. It should be unnecessary to argue regarding the admissibility of additions coming properly under the latter designation; yet it is true that in Europe such additions have not unfrequently been discovered in wines that, if left to themselves, would soon have become unsalable. It is not easy to draw the exact line between poisons proper and those substances of which the use to a certain degree, and in a certain way, may be considered admissible for the purpose of stopping undesirable fermentations in wines. There is, however, one point of view which covers the whole ground in connection with the use of wines for hygienic purposes, namely, that whatever impedes fermentations also impedes digestion, which is itself in a great degree a process of fermentation. The habitual use of wines containing antiseptics will, therefore, inevitably result in functional derangements; and this is so well understood that in Europe the extreme amounts of those allowed at all is strictly limited by law. Thus in the case of *sulphuric acid*, one of the germicides most commonly employed, partly in the form of the acid itself, but more commonly in that of plaster (sulphate of lime) added to the grapes, or to the wine itself. The tartaric acid of the wine is thus partially or wholly replaced by the sulphuric, tartrate of lime being thrown down; and thus badly made wines may be prevented from passing onward into the improper fermentations, and becoming undrinkable. *Salicylic acid* is effectual in much smaller quantities, and at one time it was thought that it would be admissible to employ it freely. But while its effects upon the human system are not apparent at first in most cases, yet the decided and unpleasant results often produced in the case of persons of weak digestion have but served to emphasize the general axiom, that we cannot, with impunity, continue to introduce into the human body substances foreign to the vegetable and animal products that have from time immemorial constituted the nutriment of mankind. If some persons are able to bear for a

¹ Report of Viticultural Work, 1883-'84, and 1884-'85, page 32.

time doses of salicylic acid that will completely stop digestion for some hours in the case of others, it is altogether unlikely that even the strongest person could continue its use indefinitely without injury. After some years of toleration, the legal prohibition of its use in articles of food or drink seems, in Europe, to be only a question of time; the more as in the case of wines, the process of "Pasteurizing" removes all legitimate reason for the longer continuation of a doubtful practice, liable to gross abuse.

In view of this fact, it is curious that its use for the conservation of must in the unfermented condition has not only been extensively introduced in this country, but the resulting beverage is especially recommended, as a healthful and harmless substitute for wine, by those who consider alcohol as necessarily harmful in any form and quantity. A few years' experience will doubtless show how unfortunate has been the choice of a substitute in this case.

And again as follows :¹

Finally, when wines are not entirely sound—and with the methods of fermentation now in vogue this is a very prevalent condition—the remedy to be applied should not lie in the use of antiseptics, sulphuring, salicylic or boracic acids, and the like, but in the simple and rational heating process devised by Pasteur, and named for him. The "Pasteurizer" should be an indispensable appliance in every wine-house; and its use, if properly understood and practiced, will at once do away with nine-tenths of all doctoring for unsoundness. The universal adoption of this simple and inexpensive expedient will save all losses now sustained in the shipment of our young wines, and will soon do away with the reproach that "California wines will not keep."

If in the face of all these facts and legitimate substitutes for medication there are those who desire to adhere to the old doctoring system, it is at least the right of those who do without them and furnish the consumers the pure product of the grape to have a legalized mode of expressing the fact on the packages.

VARIETIES.

The different kinds of wines sold can be numbered by the hundreds. They refer usually either to the country where it is produced, or of whose product it is an imitation, as Port, Sherry, Hochheimer, Madeira, &c., or to the variety of grape from which it is made, as catawba, riesling, zinfandel, &c.

No generally recognized classification is made except into *white* or *red* wines according to their color; and into *dry* or *sweet* wines according to their content of sugar. The general name of *champagne* is given to effervescing wines.

COMPOSITION OF WINE.

In countries where the production of wine is one of the leading industries, like France and parts of Germany, the composition of the wines made is very well established. Scarcely any article of consumption has been the subject of so much chemical investigation as wine. Thousands of analyses have been published, so that one is at a loss to choose among them for representative figures.

In a general way the normal constituents of a natural wine may be divided into two classes, volatile and fixed.

The volatile matters are as follows: Water, constituting from 80 to 90 per cent. of the weight; alcohol, 5 to 15 per cent.; glycerine, 2 to 8

¹ Bulletin No. 65, Univ. of Cal. Agl. Ex. Station.

per cent.; volatile acids, acetic, œnanthic, &c., constituting one-fourth to one-third of the total acidity; aldehyde, compound ethers, together with the other fragrant, indefinite constituents, which give the wine its flavor and bouquet; carbonic acid gas in small quantities in young wines.

The fixed matters are: Glucose or grape sugar in small quantities in most wines; bitartrate of potash, tartaric, malic, and phosphoric acid, partly free and partly combined with potash, lime, soda, aluminum, magnesium, iron, and manganese, of which salts phosphate of lime is the most abundant, constituting from 20 to 60 per cent. of the weight of the ash, the remainder being chiefly carbonate of potash resulting from the calcination of the bitartrate, with a little sulphate and traces of chlorides. Coloring matters: Pectin and analogous gummy matters; tannin, 1 to 2 per cent. in red wines, mere traces only existing in white.

The following table of the composition of French wines is taken from Wurtz's Dictionnaire de chimie:

Average composition of French wines.

	Alcohol by vol- ume.	In grams per liter.					Remarks.
		Extract at 100° C.	Glycer- ine.	Bitar- trate of potash.	Ash.	Total acidity as H ₂ SO ₄ .	
<i>Red wines.</i>							
Mean of French wines.	<i>Per cent.</i> 10.0	18.9	5.6-7.6	1.2-5.0	1.2-3.8	2.5	Extract (max. 40.5, min. 15.0).
Burgundy.....		16.9	5.8	1.8	2.5	
Meursault.....	10.3	Alcohol (max. 13.0, min. 9.4).
Pomard.....	11.5	Alcohol (max. 13.3, min. 10.2).
Volnay.....	10.5	Alcohol (max. 12.6, min. 7.3).
Richebourg, ordi- nary.	11.2	23.6	Wines six months old.
Petits Burgundy...	7.8	15.6	Wines one year old.
Beaujolais.....	10.4	20.7	2.17	Wines three years old.
Bordeaux, ordinary.	9.4	7.1	2.3	1.6-3.0	Alcohol (max. 10.9 min. 7.9).
Bordeaux, superior.	9.1	16.4	2.15	Alcohol (max. 9.7, min. 7.3).
Vins de l'Herault, (not plastered).	10.1	19.0	6.5-7.6	2.2	1.7-3.5	2.5-5.6	Extract (max. 23.0, min. 16.0).
Vins de l'Herault, (plastered).	10.3	20.0-25.0	3.2-4.6	4.9-5.1	
Aramon (plastered).	10.0	24.0	2.95	4.2	
Beaux Narbonnes (1876).	12.4	
Narbonnes (not plastered).	10.8	18.8	2.0-3.0	Alcohol (max. 14.0, min. 8.4).
Petits Narbonnes (1876).	7.8	
Rousillons de table (1875).	14.8	2.4	2.66	
Vins de la Marne ..	11.4	24.1	1.8-5.3	Alcohol (max. 14.0, min. 6.7).
Vins de Cahors	10.7	Alcohol (max. 14.3, min. 8.9).
Vins de la Haute- Garonne.	9.8	23.8	1.4	1.4-2.0	Alcohol (max. 12.4, min. 7.5).
							Extract (max. 28.8, min. 18.9).
<i>White wines.</i>							
Chablis (6 months) ..	9.7	14.0	2.51	
Bordeaux.....		4.5	
Picpoul.....	11.5	18.2	3.5	

The following table of the average composition of wines of various
u. s. w.:

Average composition of the wines of all

Country.	No. of wines analyzed.	Specific gravity.				Alcohol by volume.				Acids, as tartaric.			
		No. of estimations.	Maximum.	Minimum.	Mean.	No. of estimations.	Maximum.	Minimum.	Mean.	No. of estimations.	Maximum.	Minimum.	Mean.
America (Virginia)....	12	12	1.0117	0.9875	0.9956	12	12.69 ¹	8.56 ¹	10.62 ¹	12	1.02	0.52	0.671
Australia.....	5	5	5	18.0	14.1	15.5	5	0.510	0.450	0.494
Africa.....	4	1	0.9938	2	20.3	18.0	19.1	3	0.370	0.224	0.275
Minor Asia.....	8	7	1.0892	1.0051	1.0325	8	18.0	13.0	14.3
Crimea.....	31	31	1.0011	0.9875	0.9942	31	16.93	9.08	12.80	31	0.854	0.350	0.592
Greece.....	9	7	1.0254	0.9909	1.0109	9	18.0	12.4	15.4
Spain.....	9	7	1.0700	1.0370	1.0593	8	16.1	12.5	14.6	1	0.339
Italy.....	40	40	1.0879	0.8934	1.0019	40	21.95	8.40	13.86	40	1.0425	0.276	0.674
Sicily.....	86	86	1.0976	0.9895	1.0225	86	27.15	13.70	18.08	86	0.8352	0.380	0.591
France.....	60	27	1.0019	0.9910	0.9952	47	14.0	6.5	9.9	40	0.970	0.190	0.506
Switzerland.....	68	14	0.9930	0.9380	0.9904	68	13.1	6.0	9.0	68	0.750	0.370	0.528
Austria.....	523	488	1.0797	0.9896	0.9941	503	18.8	7.5	11.0	499	0.995	0.110	0.588
1. Lower Austria.....	122	113	1.0034	0.9898	0.9857	122	15.7	8.6	13.0	113	0.910	0.434	0.632
2. Styria.....	95	80	1.0797	0.9908	0.9987	86	15.3	8.1	12.0	92	0.995	0.116	0.661
3. Tyrol.....	4	4	0.9966	0.9952	0.9959	4	8.3	7.5	7.9	3	0.612	0.461	0.523
4. Mähren.....	4	4	0.9942	0.9932	0.9937	4	11.7	9.8	10.6	4	0.727	0.496	0.577
5. Bohemia.....	19	19	0.9994	0.9921	0.9950	19	14.1	9.5	11.6	19	0.806	0.426	0.613
6. Carniola.....	11	11	0.9963	0.9939	0.9952	11	10.5	8.5	9.2	11	0.811	0.465	0.601
7. Hungary.....	154	145	1.0201	0.9910	0.9960	148	18.8	8.4	12.2	144	0.953	0.349	0.635
8. Transylvania.....	4	4	0.9928	0.9876	0.9904	4	12.8	10.5	11.7	4	0.498	0.433	0.469
9. Slavonia.....	38	38	1.0012	0.9921	0.9944	38	15.1	9.3	11.3	38	0.928	0.573	0.697
10. Croatia.....	72	70	1.0086	0.9910	0.9943	66	13.6	7.5	10.5	70	0.944	0.449	0.687
11. Dalmatia.....	1	1	0.9950	1	10.7	1	0.480
Germany.....	616	224	1.0833	0.9960	0.9954	615	16.0	5.7	10.1	544	1.354	0.050	0.599
1. Saxony.....	2	1	0.9975	2	9.0
2. Silesia.....	1	1	0.9976	1	6.5
3. Mosel-Saar.....	11	4	0.9977	0.9930	0.9946	11	14.2	6.7	10.6	7	0.660	0.560	0.612
4. Ahrgegend.....	11	11	0.9930	0.9915	0.9944	11	11.2	7.9	9.8	11	0.559	0.390	0.485
5. Rheingau.....	57	42	1.0323	0.9960	0.9956	56	16.0	8.2	11.3	48	0.750	0.332	0.545
6. Rheingau-Bergstr.....	30	26	0.9996	0.9910	0.9950	31	11.7	7.9	10.0	25	0.810	0.359	0.592
7. Palatinate.....	62	41	1.0034	0.9868	0.9955	62	13.3	6.8	10.7	24	0.779	0.350	0.541
8. Franconia.....	78	69	1.0833	0.9762	0.9940	78	13.3	6.5	10.6	73	1.354	0.453	0.850
9. Württemberg.....	27	13	0.9982	0.9937	0.9956	27	15.5	7.0	11.4	23	0.830	0.350	0.618
10. Baden.....	336	16	0.9986	0.9935	0.9945	336	15.6	5.7	11.2	333	1.087	0.050	0.546
11. Alsace.....	12	37	0.9950	0.9860	0.9907	37	12.06	6.20	10.28	37	0.705	0.332	0.543
<i>German kinds.</i>													
Riesling.....	85	17	1.0025	0.9911	0.9948	85	14.7	8.8	11.3	78	0.960	0.080	0.659
Traminer.....	40	6	0.9997	0.9762	0.9921	40	14.3	8.7	11.8	39	0.907	0.330	0.588
Ruländer.....	20	20	14.6	9.4	11.0	19	0.780	0.420	0.562
Gutedel.....	33	5	0.9939	0.9927	0.9934	33	12.1	8.9	10.3	32	0.705	0.274	0.531
Weissherbst.....	15	2	0.9966	15	13.5	8.3	11.2	15	0.640	0.286	0.526
Burgunder.....	26	2	0.9950	26	13.4	9.0	11.2	26	0.756	0.429	0.574
Clevnerblau.....	30	6	0.9982	0.9933	0.9955	30	15.2	8.2	11.5	30	0.907	0.416	0.595

¹ Per cent. by weight.

origin consumed in Germany is taken from König's Nahrungsmittel, countries (*Wagenmann and König*).

Sugar.				Extract.				Tannin and coloring matter.				Ash.			
No. of estimations.	Maximum.	Minimum.	Mean.	No. of estimations.	Maximum.	Minimum.	Mean.	No. of estimations.	Maximum.	Minimum.	Mean.	No. of estimations.	Maximum.	Minimum.	Mean.
	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>		<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>		<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>		<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>
12	3.703	0.031	0.724	12	6.41	1.41	2.581	12	0.019 ²	0.02 ²	0.009 ²	12	0.30	0.12	0.175
5	3.500	0.840	1.486	5	4.800	2.600	3.240								
				2	5.510	4.190	4.580					1			0.428
				16	230	2.400	6.292								
5	3.403	0.040	1.563	33	4.515	1.353	2.516	9	0.503	0.143	0.207	33	0.382	0.153	0.227
				9	4.800	1.400	3.418								
7	14.700	9.900	11.900	8	18.780	14.400	16.520								
82	20.976	0.179	3.630	407	21.886	0.746	3.259	82	0.339	0.003	0.073	407	0.743	0.057	0.214
				86	27.940	0.746	8.442					86	0.857	0.185	0.363
3	0.180	0.109	0.159	40	12.600	1.080	3.036	14	0.229	0.186	0.207	17	0.252	0.174	0.216
14	0.120	0.025	0.063	14	2.689	1.293	1.878					14	0.502	0.134	0.249
9	6.100	1.200	2.647	495	23.140	0.870	2.422	15	0.194	0.109	0.139	186	0.323	0.077	0.188
				122	6.320	1.790	2.650	10	0.194	0.109	0.139	17	0.311	0.162	0.241
2			3.360	77	23.140	1.090	3.730					47	0.305	0.087	0.165
				4	1.850	1.670	1.750					4	0.228	0.154	0.184
				4	2.280	1.880	2.010								
				19	3.910	1.850	2.620					7	0.297	0.128	0.197
1			1.620	11	2.510	1.480	2.000					11	0.269	0.120	0.182
6	6.100	1.200	2.960	149	10.700	0.870	3.050	5	0.175	0.111	0.141	36	0.288	0.077	0.192
				4	2.170	1.450	1.750								
				38	3.890	1.510	2.360					14	0.286	0.145	0.191
				66	5.000	1.210	2.140					49	0.323	0.111	0.167
				1			2.600					1			0.170
409	8.628	0.010	0.470	386	10.555	0.520	2.502	41	0.272	0.076	0.145	94	0.314	0.108	0.194
1			1.000	2			3.000								
				1			2.100								
7	0.520	0.120	0.241	9	2.500	1.500	1.892					1			0.203
11	0.674	0.056	0.159	11	2.885	2.137	2.671	10	0.272	0.099	0.214	11	0.261	0.099	0.213
30	8.628	0.080	0.964	56	10.555	1.640	3.087	16	0.261	0.091	0.141	29	0.314	0.120	0.215
15	1.500	0.100	0.410	31	4.100	1.040	2.588	13	0.235	0.091	0.148	17	0.275	0.125	0.213
43	3.500	0.610	0.560	49	7.300	1.710	2.236	2			0.076	22	0.205	0.108	0.137
6	0.154	0.081	0.138	76	9.445	1.112	3.165								
18	1.400	0.080	0.305	17	2.920	2.750	2.255								
279	4.540	0.046	0.442	134	3.545	0.520	1.730					14	0.255	0.163	0.183
29	0.188	0.013	0.087	36	2.389	1.228	1.784					25	0.387	0.105	0.225
75	4.540	0.091	0.458	36	9.940	0.790	2.462					5	0.190	0.146	0.170
33	4.160	0.046	0.480	16	3.612	0.520	2.200					2			0.183
20	1.500	0.050	0.239	14	2.700	1.320	1.848								
26	0.610	0.077	0.124	24	2.735	1.420	1.781					5	0.183	0.160	0.172
12	2.100	0.070	0.586	4	2.290	1.770	2.012					2			0.190
23	0.275	0.087	0.119	14	2.706	1.360	2.170	1			0.272	2			0.229
28	3.330	0.076	0.871	12	4.167	1.390	2.364	3	0.272	0.190	0.230	3	0.253	0.181	0.212

² Given as tannic acid.

COMPOSITION OF AMERICAN WINES.

The earliest analyses of American wines on record were made by Merrick,¹ in 1875, comprising six varieties of California wines.

In October of the same year Mallet and Cooper² published analyses of twelve samples of Virginia wines. The mean of these analyses is given in the table from König.

The work of Professor Hilgard on California wines began in 1880 and has continued down to the present day, the results being published in the Bulletins of the Station. These publications include extensive series of analyses, which afford a most valuable index of the composition of California wines, especially as many of the analyses were made on wines manufactured in the laboratory and hence known to be absolutely pure. A standard of composition could very properly be established from them, and a limit for the amount of each constituent present in pure wines, by which the addition of alcohol, water, sugar, &c., in sophisticated wines could be detected. The number of different determinations made on each sample is not very large, unfortunately, including only the more important constituents. I give below tables of the entire work done on wines known to be pure :

Composition of wines made at the Viticultural Laboratory, 1884.

Variety.	Body.	Alcohol.		Tan- nin.	Acid as tar- taric.	Ash.
		By weight.	By volume.			
<i>Bordeaux type.</i>						
Malbeck	2.68	<i>P. ct.</i> 8.34	<i>P. ct.</i> 10.42	<i>P. ct.</i> .100	<i>P. ct.</i> .450	<i>P. ct.</i> .393
Cabernet Franc	2.84	9.63	12.00	.035	.480	.432
Do	2.13	8.48	10.58	.070	.607	.293
Cabernet Sauvignon	3.19	9.92	12.36	.079	.540	.447
Merlot	2.44	9.20	11.42	.065	.467	.394
Verdot	2.77	9.78	11.82	.071	.438	.409
Tannat	2.69	7.46	8.92	.171	.633	.353
Beclan	2.64	8.84	11.00	.053	.381	.260
Carignane	2.18	7.92	9.90	.063	.527	.285
Grossblau	2.10	9.20	11.42	.065	.572	.252
<i>Burgundy type.</i>						
Black Burgundy	2.77	9.34	11.64	.188	.765	.277
Black Pinot	2.11	6.42	8.00	.133	.750	.310
Meunier	2.55	10.07	12.55	.025	.525	.419
Do	2.44	6.95	8.64	.055	.622	.285
Zinfandel	2.36	7.85	9.75	.036	.450	.425
Do	1.93	7.43	9.25	.080	.633	.200
Do						
Do						
Do						
<i>Southern French and Italian types.</i>						
Trousseau	2.66	11.23	13.73	.075	.485	.379
Do	2.28	9.92	11.64	.050	.474	.349
Petite Sirah	2.65	10.07	12.54	.092	.401	.406
Do	2.79	10.81	13.27	.108	.293	.339
Serine	2.49	8.48	10.58	.063	.510	.400

¹ Amer. Chemist 6, 85.

² Chem. News 32, 160.

Composition of wines made at the Viticultural Laboratory, 1884.

Variety.	Body.	Alcohol.		Tan- nin.	Acid as tar- taric.	Ash.
		By weight.	By volume			
<i>Southern French and Italian types—Continued.</i>						
Mondeuse.....	2.64	<i>P. ct.</i> 9.56	<i>P. ct.</i> 11.89	<i>P. ct.</i> .173	<i>P. ct.</i> .498	<i>P. ct.</i> .242
Do.....	2.84	9.92	12.27	.141	.405	.362
Cinsaut.....	2.60	10.44	12.90	.070	.490	.375
Aramon.....	2.32	9.05	10.73	.065	.496	.356
Mourastel.....	2.60	7.92	9.88	.054	.553	.291
Grenache.....	1.67	8.48	10.58	.105	.480	.281
Do.....	1.93	7.43	9.27	.065	.532	.277
Petit Bouschet.....	2.44	8.84	11.00	.089	.583	.234
Do.....	2.87	9.27	11.55	.117	.493	.369
Clairette Rouge.....	2.72	9.92	12.36	.063	.510	.293
Barbera.....						
Lenoir.....	3.00	8.13	10.16	.060	.630	.376
Do.....	3.71	8.98	11.17	.075	.582	.511
Blau-Elbling.....	1.93	8.27	10.25	.040	.585	.340
Mission.....						
Black Prince.....						
<i>Dry white wine varieties.</i>						
Semillon.....						
Do.....	1.93	9.92	12.36		.450	.175
Sauvignon Blanc.....						
Do.....	2.10	9.92	12.36		.453	.157
Do.....	2.18	10.26	12.75		.533	.192
Muscadelle du Bordelais (loose bunches).....	2.13	11.46	14.00		.432	.208
Muscadelle du Bordelais (compact bunches).....	2.44	10.81	13.27		.498	.342
Folle Blanche ("Tannat").....	1.66	9.92	12.36		.593	.214
Folle Blanche.....	1.80	7.43	8.75		.570	.235
Burger.....	2.05	8.41	10.50		.600	.260
Do.....	1.65	8.55	10.63		.528	.181
Roussanne.....	1.36	10.54	13.00		.468	.260
Marsanne.....	1.88	9.20	11.46		.563	.176
Clairette Blanche.....	1.85	10.54	13.00		.428	.212
"Pecoui Touar" (?).....	1.66	9.27	11.58		.573	.234
<i>Sherry and Madeira varieties.</i>						
Pedro Jimenes.....	1.18	9.63	12.00		.555	.390
Palomino.....	1.85	11.08	13.50		.528	.250
Peruno.....	1.95	9.92	12.50		.519	.306
Mantuo de Pilas.....	1.32	7.85	9.82		.525	.265
Mourisco Branco.....						
Beba.....	2.10	11.69	14.27		.503	.372
Verdelho.....	2.82	12.39	15.20		.417	.330
Boal Madeira.....	2.00	9.27	11.58		.660	.264
Ugni Blanc.....	2.00	8.91	11.10		.510	.299
Malmsey.....	1.60	7.92	9.91		.543	.160
Malaga.....	1.69	8.13	9.82		.217	.240
<i>Port wine varieties.</i>						
Tinta Cao.....						
Tinta Madeira.....						
Mourisco Preto.....						
Tinta Amarella (not fully ripe).....	2.36	9.05	11.27	.058	.501	.446
Tinta Amarella (fully ripe).....	2.39	9.56	11.91	Slight	.525	.470
Moretto.....						
Bastardo.....	3.12	10.35	12.83	.062	.507	.564

Analyses of wines made at the Viticultural Laboratory, 1885.

Variety.	Body.	Alcohol.		Tannin.	Acid.		Ash.
		By weight.	By volume.		Tartaric.	Volatile.	
<i>Bordeaux type.</i>							
		<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>P. ct.</i>
Malbeck ¹							
Pfeffer's Cabernet	2.99	8.84	11.00	.123	.846		.273
Do	2.69	10.54	13.00	.101	.516		.290
Do	2.69	8.13	10.17	{ (1).179	.486		.366
Merlot	2.69	9.20	11.45	{ (2).153	.420		.310
				.21			
<i>Burgundy type.</i>							
Burgundy	3.07	9.63	12.00	.071	.555		.296
Bl. Pinot or Tree Burgundy	2.84	8.91	11.09	.110	.600		.315
Black Pinot	2.20	7.37	9.18	.040	.525		.214
Franc Pinot ¹							
Meunier ¹							
Zinfandel ¹							
Do	2.69	8.84	11.00	.086	.576		.290
Do	2.69	11.46	14.00	.133	.417		.280
Charbono ¹							
Do	2.69	7.09	8.85	.112	.495		.270
Do	2.26	7.78	9.73	.160	.420		.290
Do	2.18	7.23	9.00	.209	.547		.270
Do	1.80	6.42	8.08	.130	.502		.330
<i>Southern French and Italian type.</i>							
Trousseau ¹							
Mataro	1.98	7.23	9.00	.080	.487		.290
Teinturier	3.07	8.06	10.08	.184	.555		.340
Kadarka	1.80	6.82	8.54	.070	.450		.236
Blend: 90 per cent. Zinfandel, 10 per cent. Charbono	2.84	8.84	11.00	.108	.576		.213
<i>Dry white varieties.</i>							
Chauché Gris	1.80	10.54	13.00		.351		.250
Verdal	1.80	6.42	8.07		.517		.188
One-third Golden Chasselas, two-thirds Burger	1.80	9.63	12.00		.615		.217
Burger	2.69	9.63	12.00		.675		.450
Do	1.93	8.13	10.16		.645		.192
Do	1.68	8.84	11.00		.540		.183
Do	1.52	4.76	6.00		.480		.225
Franken Riesling ²	3.82	8.48	10.58		.600	.366	.270
Riesling	1.80	9.05	11.27		.352		.160
Colombar	2.26	8.84	11.00		.591		.280
Zinfandel, first crop	2.30	11.23	13.73		.481		.244
Zinfandel, second crop	2.15	10.54	13.00		.525		.140
Zinfandel, second crop, "filtered"	2.60	10.54	13.00		.576		.187
Zinfandel, 97½ per cent.; Verdal, 2½ per cent.	2.15	10.54	13.00		.503		.191
Burger, 50 per cent.; Zinfandel 50 per cent.	2.10	11.08	13.54		.713		.184

¹ Not enough for wine-making.

² Sugar = 65 per cent.

Composition of wines made at Viticultural Laboratory, 1886.

Variety.	Alcohol.		Tannin.	Acid as tartaric, at pressing.
	By weight.	By volume.		
<i>Bordeaux type.</i>				
	<i>Per cent.</i>	<i>Per cent.</i>	<i>P. ct.</i>	<i>P. ct.</i>
Malbeck	8.27	10.36	.132	.62
Cabernet Franc.....	9.78	12.18	.264	.45
Cabernet Sauvignon.....	8.48	10.58	.226	.52
Pfeffer's Cabernet	9.05	11.27	.190	.42
Do	8.34	10.42	.208	.73
Gros Verdot	7.92	9.90	.166	.79
Tannat	9.70	12.09	.316	.77
Beclan	8.34	10.42	.090	.60
Charbono	7.23	9.00	.251	.53
Do	7.64	9.54	.186	.60
Carignane	9.27	11.55	.172	.69
Do				
Grossblanc	6.95	8.70	.169	.81
Do	7.09	8.85	.195	.72
Black Hamburg.....	7.23	9.00	.108	.52
West's St. Peter's (?).....	8.84	11.00	.276	.57
<i>Burgundy type.</i>				
Burgundy.....	9.99	12.46	.226	.43
Crabb's Black Burgundy.....	7.64	9.54	.220	.47
Do				
"Burgundy" (Chauché Noir ?).....	9.92	12.36	.074	.41
Chauché Noir.....	8.48	10.58	.106	.38
Petit Pinot (?).....	9.92	12.36	.076	.53
Pinot (?).....	10.81	13.27	.153	.35
Pinot St. George.....	9.92	12.36	.112	.53
Meunier	9.49	11.82	.133	.55
Do	9.92	12.36	.193	.40
Zinfandel	9.92	12.36	.104	.54
Do	11.62	14.20	.140	.54
Do	9.20	11.45	.091	.59
Do	8.48	10.58	.169	.94
Do	7.23	9.00	.148	.55
Zinfandel, second crop.....	7.78	9.73	.103	.71
Zinfandel (?) (Row 31).....	7.09	8.85	.151	.64
<i>Southern French and Italian types.</i>				
Trousseau	10.63	13.09	.090	.45
Ploussard	9.56	11.91	.051	.51
Sirah	8.84	11.00	.107	.65
Mondense	7.64	9.54	.127	.78
Do	7.99	10.10	.317	.64
Do				
Cinsaut	7.78	9.73	.153	.90
Do	7.99	10.00	.110	.52
Barbera	9.20	11.45	.153	.85
Teinturier	9.85	12.27	.278	.73
Do	8.34	10.42	.114	.64
Gamay Teinturier.....	8.48	10.58	.169	.90
Nebbiolo Bourgu.....	8.13	10.17	.172	.82
Nebbiolo fino.....	8.91	11.09	.215	.86
Fresa	6.49	8.15	.160	1.12
Blend: Barbera; Herbemont, one-third; Fresa.....	7.37	9.18	.256	.63
<i>American type.</i>				
Herbemont	6.89	8.61	.095	.74
Do	7.43	9.27	.146	.50
Californica	7.09	8.85	.179	.61
Isabella regia				
<i>Dry white wine varieties.</i>				
Semillon	7.23	9.00		.55
Sauvignon Vert.....	7.23	9.00		.53
Folle Blanche.....	7.78	9.73		.59
Burger	8.48	10.56		.47

Composition of wines made at Viticultural Laboratory, 1886—Continued.

Variety.	Alcohol.		Tannin.	Acid as tartaric, at pressing.
	By weight.	By volume.		
<i>Dry white wine varieties—Continued.</i>				
Burger.....	<i>Per cent.</i> 4.14	<i>Per cent.</i> 5.23	<i>P. ct.</i>	<i>P. ct.</i> .81
Do.....	7.99	10.00		.33
Kleinberger.....	6.95	8.70		.43
Marsanno.....	8.48	10.58		.56
Clairette Blanche.....	7.09	8.85		.47
Do.....				
Do.....	7.57	9.45		.35
Do.....	8.48	10.58		.47
"Pecoui Tonar".....	7.09	8.85		.45
Franken Riesling.....				
Do.....	7.23	9.00		.50
Riesling.....	7.37	9.18		.36
Riesling (?).....	9.20	11.45		.55
Johannisberger Riesling.....	8.13	10.17		.60
Chasselas de Fontainebleau.....	6.89	8.61		.41
Chauché Gris.....	8.27	10.36		.45
"Gray d Ischia," Chauché Gris.....	8.98	11.18		.38
Seedless Sultana.....	8.27	10.36		.51
White Muscateller.....	8.70	10.83		.44
Blau-Elbling.....	8.13	10.17		.54
White Zinfandel.....	9.05	11.27		.41
Cinsaut.....	9.27	11.55		.48
Verdal.....	8.48	10.58		.25
Do.....				
<i>Sherry and Madeira varieties.</i>				
Pedro Jimenes.....	7.92	9.90		.42
Palomino.....				
Palomino ("Golden Chasselas").....	6.35	8.00		.54
Mantuo de Pilas.....	7.78	9.73		.45
Do.....	8.48	10.58		.35
Mourisco Branco.....	10.07	12.54		.33
Verdelho.....				
Muscat of Alexandria.....	11.62	14.20		.42
Do.....	9.56	11.91		.36
Do.....				
West's White Prolific, with some smaller grapes.....	7.78	9.73		.72
West's White Prolific.....	7.78	9.73		.71
"White St. Peter's".....	8.48	10.58		.45

As this work seems to be very important as establishing the average composition of pure wines made in California, I have prepared from Professor Hilgard's reports a table showing the maximum, minimum, and mean composition of the pure wines analysed, as well as of the wines which were made outside and sent in to the laboratory for analysis:

Maximum, minimum, and mean composition of California wines, as shown by the analyses made at the California State Viticultural Laboratory.

	No. of samples analyzed.	Alcohol by weight.			Body or extract.			Total acids as tartaric.			Ash.		
		Maximum.	Minimum.	Mean.	Maximum.	Minimum.	Mean.	Maximum.	Minimum.	Mean.	Maximum.	Minimum.	Mean.
<i>Pure wines made at laboratory in 1884.</i>													
Bordeaux type	10	<i>P. ct.</i> 9.92	<i>P. ct.</i> 7.46	<i>P. ct.</i> 8.81	<i>P. ct.</i> 3.19	<i>P. ct.</i> 2.10	<i>P. ct.</i> 2.57	<i>P. ct.</i> .633	<i>P. ct.</i> .381	<i>P. ct.</i> .510	<i>P. ct.</i> .447	<i>P. ct.</i> .252	<i>P. ct.</i> .352
Burgundy type	6	10.07	6.42	9.01	2.77	1.93	2.36	.765	.450	.624	.425	.200	.319
Southern French and Italian type	18	11.23	7.43	9.26	3.71	1.67	2.56	.585	.393	.506	.511	.234	.343
Dry white wine varieties	13	11.46	7.43	9.71	2.44	1.36	1.90	.600	.428	.516	.342	.157	.219
Sherry and Madeira varieties	10	12.39	7.85	9.68	2.82	1.18	1.85	.660	.217	.498	.290	.160	.288
Port wine varieties	3	10.35	9.05	9.65	3.12	2.36	2.62	.525	.501	.511	.564	.446	.493
<i>Pure wines made at laboratory in 1885.</i>													
Bordeaux type	4	10.54	8.13	9.18	2.99	2.69	2.76	.846	.420	.567	.366	.273	.310
Burgundy type	9	11.46	6.42	8.30	3.07	1.80	2.49	.600	.417	.515	.330	.214	.276
Southern French and Italian type	4	8.81	6.82	7.74	3.07	1.80	2.42	.576	.450	.517	.340	.213	.270
Dry white wine varieties	15	11.23	4.76	9.22	3.82	1.52	2.16	.713	.351	.544	.450	.140	.224
<i>Pure wines made at laboratory in 1886.</i>													
Bordeaux type	15	9.78	6.95	8.28810	.420	.620
Burgundy type	16	11.62	7.09	9.21940	.350	.540
Southern French and Italian type	15	10.63	6.49	8.48	1.120	.450	.730
Dry white wine varieties	25	9.27	4.14	7.86810	.250	.480
Sherry and Madeira varieties	10	11.62	6.35	8.58720	.330	.480
American type	3	7.43	6.89	7.14740	.500	.620
<i>Wines sent to laboratory for analysis, 1884-'85.</i>													
Reds	20	10.60	7.64	9.63	3.62	2.05	2.87	.750	.395	.563	.534	.219	.340
Whites	5	10.81	8.98	9.80	3.87	2.05	2.67	.527	.397	.473	.367	.255	.303
<i>Wines sent to laboratory for analysis, 1885-'86.</i>													
Reds	55	16.42	7.99	10.48	13.77	2.10	4.30	.879	.225	.554	.470	.230	.324
Whites	16	12.39	8.84	10.82	4.20	1.80	2.66	.750	.210	.473	.300	.170	.261
Zinfandel wines analyzed, 1879-'85.	45	12.39	7.43	10.55	8.64	1.46	2.89	.873	.337	.573	.546	.154	.301

In the year 1880 a large number of samples of wine were purchased in the market of Washington and analyzed by the Department of Agriculture. The work was under the charge of the late Henry B. Parsons, one of the most competent analysts ever in the service of the Department. The results are published in the Annual Report for 1880, forming part of the Chemists' report for that year. I have thought it proper to reproduce them here, as the results include a large number of determinations on each sample, and give very important information in regard to the composition of American wines as they are supplied to the consumer.

I.—DRY RED WINES.

No.	Name.	Specific grav- ity.	Alcohol by weight.	Alcohol by volume.	Total residue.	Total ash.	Glucose.	Total acid, as tartaric.	Fixed acid, as tartaric.	Volatile acid, as acetic.	Maker.
		Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	
3	Virginia Claret.	.9941	9.61	12.65	2.03	0.193	0.13	0.725	0.393	0.266	Monticello Wine Company.
16	Virginia Claret, Concord, 1879.	.9953	8.83	11.08	2.10	.174	Trace	.709	.452	.206	Do.
17	Virginia Clinton, 1879.	.9950	9.82	12.31	2.36	.238	Trace	.784	.513	.217	Do.
18	Cynthiana, 1880.	.9969	10.24	12.87	2.95	.283	.09	.647	.376	.217	Do.
19	Alvey, 1880.	.9931	9.77	12.22	2.13	.174	Trace	.680	.498	.146	Do.
20	Norton's Virginia, 1879.	.9937	10.21	12.77	2.88	.298	do	.772	.377	.316	Do.
22	Ives' Seedling, 1879.	.9914	8.68	10.82	2.18	.247	do	.793	.512	.169	Do.
22	Ives' Seedling, 1879.	.9906	8.36	8.01	2.62	.275	do	.825	.381	.355	Do.
32	Norton's Virginia, 1875.	.9983	8.26	10.38	2.95	.295	do	.762	.481	.385	Do.
33	Concord, 1873.	1.0011	7.25	7.25	2.80	.273	do	.704	.395	.247	Do.
33	do	.9970	7.65	9.62	2.53	.359	None	.722	.316	.325	Do.
33	Clinton, 1872.	.9982	5.71	7.17	2.07	.192	Trace	.902	.292	.488	Do.
37	do	.9961	7.93	9.95	2.19	.196	None	.798	.391	.326	Do.
34	St. Julien.	.9959	7.14	8.96	1.99	.210	do	.587	.247	.272	Do.
35	La Rose.	.9987	7.91	9.95	2.61	.268	do	.766	.400	.293	Do.
35	Claret, 1874.	.9988	9.02	11.35	2.69	.236	do	.826	.411	.332	Do.
59	Concord 1880.	.9933	8.72	10.91	2.38	.185	.45	.619	.332	.230	C. Xander.
60	Clinton, 1880.	.9920	10.00	13.62	2.49	.165	.30	.620	.302	.254	Do.
61	Ives, 1880.	.9925	8.65	10.82	2.17	.192	.20	.680	.363	.254	Do.
62	Norton's Virginia, 1880.	.9941	8.99	11.26	2.38	.222	Trace	.662	.308	.283	Do.
63	Ives and Clinton, 1880.	.9920	9.62	12.65	2.17	.183	Trace	.635	.372	.210	Do.
64	Ives and Clinton, 1879.	.9936	9.28	11.61	2.49	.208	do	.709	.386	.258	Do.
65	Concord and Clinton, 1879.	.9943	9.76	12.22	2.38	.202	do	.754	.398	.285	Do.
84	Franklin, 1868.	.9955	8.77	11.00	2.32	.166	do	.859	.362	.398	J. Julius Hincke.
87	Kohlunk, 1868.	.9935	5.72	10.91	1.06	.151	do	.830	.323	.406	Do.
74	Ruby Claret, 1875.	.9910	9.73	12.13	2.22	.156	do	.726	.475	.201	J. H. Bannhr.
75	Ruby Claret, 1876.	.9917	10.30	12.87	2.06	.186	do	.696	.463	.186	Do.
76	Ruby Claret, 1877.	.9927	9.56	11.96	1.94	.165	do	.695	.438	.206	Do.
77	Ruby Claret, 1878.	.9902	11.82	14.74	1.87	.140	do	.687	.373	.395	Do.
78	Ruby Claret, 1879.	.9918	10.15	12.63	1.84	.154	do	.650	.399	.201	Do.
79	Ruby Claret, 1880.	.9922	10.75	13.43	1.82	.183	do	.544	.372	.138	Do.
80	Cleveland, 1876.	.9984	6.99	8.80	2.15	.250	do	.511	.264	.198	Do.
81	Cynthiana, 1876.	.9939	7.94	9.95	2.28	.208	do	.770	.347	.193	Do.
82	Franklin, 1876.	.9945	8.64	10.82	2.01	.180	do	.724	.317	.302	Do.
83	Norton's Virginia, 1877.	.9914	10.38	12.96	1.87	.166	do	.635	.287	.278	Do.
155	Ives' Seedling, 1880.	.9925	9.43	11.79	1.98	.238	do	.568	.287	.225	Poeschel, Scherer & Co.

157	Cynthiana, 1880.....	9952	9.26	11.61	2.66	343do.....	561	289	218	Toeschel, Scherer & Co.
158	Clinton, 1880.....	9994	12.21	15.21	2.20	285do.....	540	309	185	Do.
160	Virginia Seedling, 1880.....	9950	3.89	12.40	3.00	365do.....	494	302	134	Do.
162	Concord, 1880.....	9913	10.38	12.96	1.76	233	Trace.....	496	302	155	Do.
139	Sonoma Red Mission, 1879.....	9968	7.99	10.03	2.42	428	None.....	722	301	337	Gretsch & Mayer.
147	Sonoma Red Zinfandell, 1879.....	9962	7.80	9.78	2.43	555	Trace.....	693	391	242	Do.
154	Virginia Concord, 1879.....	9965	7.60	9.54	2.11	231do.....	753	421	266	Do.
89	Concord.....	9944	8.43	10.56	1.85	130do.....	648	272	301	Isaac Cook.
90	Virginia Seedling.....	9938	9.62	12.05	2.06	218do.....	664	296	350	Do.
91	Fine Claret.....	9937	9.21	11.52	1.97	189do.....	664	287	302	Do.
92	Burgundy.....	9940	9.21	11.52	2.03	210do.....	664	242	178	Do.
120	"I and N" 1880.....	9949	8.22	10.30	2.09	318	None.....	754	362	314	Wehrle, Werk & Co.
121	Norton, 1880.....	9888	12.14	15.12	2.32	176	Trace.....	516	258	206	Do.
122	Ives, 1880.....	9961	6.50	8.97	1.72	198	None.....	602	346	205	Do.
124	Concord, 1880.....	9947	8.14	10.21	2.17	209do.....	618	347	217	Do.
15	Black Rose.....	9920	9.86	12.31	1.94	170	Trace.....	707	287	375	Charles Saalhuann.
56	"Old Dominion".....	9970	6.16	7.74	1.65	169do.....	736	277	344	C. A. Heineken.
126do.....	9967	7.01	8.80	2.39	152	Trace.....	782	391	313	Do.
127	"Prince William".....	9945	10.20	12.77	3.16	297do.....	699	317	306	Do.
114	Mission Claret.....	9975	7.39	9.29	2.40	532do.....	917	271	517	B. Dreyfus & Co.
115	Red Zinfandell.....	9960	9.04	11.35	2.67	280do.....	768	277	393	Do.
171	Norton's Seedling, 1878.....	9945	9.34	11.70	2.36	261do.....	685	356	271	H. T. Dewey & Son.
172	Concord and Norton, 1879.....	9986	7.58	9.54	2.39	223	None.....	997	646	281	Do.
66	Zinfandell 1878.....	9957	8.21	10.30	2.45	213	Trace.....	825	437	310	Dresel & Co.
67	Zinfandell, 1879.....	9963	8.63	11.08	2.68	188do.....	798	376	338	Do.
37	Zinfandell.....	9947	9.83	12.31	2.56	376do.....	814	323	393	George Hamlin & Co.
38	California Claret.....	9964	8.41	10.56	2.43	326do.....	903	331	458	Do.
4	California Zinfandell.....	9930	10.38	13.24	2.21	237do.....	726	266	368	Vineyard Wine Company, New Jersey.
11	Concord Bouquet.....	9928	9.84	12.31	2.18	141do.....	741	272	375	Do.
12	Red Wine.....	1.0069	9.07	11.44	3.90	185do.....	790	402	310	Do.
97	Dry Concord, 1880.....	9913	9.44	11.79	1.66	098	Trace.....	681	272	327	W. J. Green, Tokay Vineyards.
136	Ives, 1874.....	1.0006	5.69	7.17	2.68	193do.....	764	420	275	White Elk Vineyards.
180	Red Concord.....	9953	7.34	9.21	1.87	197	None.....	838	377	289	Fritz Baier.
181	Clinton.....	9944	9.27	11.61	2.50	217do.....	778	483	236	Do.
182	Norton's.....	9933	10.66	13.34	2.70	358do.....	634	323	249	Do.

II.—DRY WHITE WINES.

1	California White Hoek.....	9892	13.94	17.37	2.62	0.243	0.09	0.855	0.231	0.311	Lenk Wine Company.
5	Missouri.....	9908	10.68	13.34	1.45	204do.....	547	227	256	Perkins, Stern & Co.
6	Riesling.....	9902	10.55	13.05	1.62	181do.....	620	227	314	Monticello Wine Company.
8	California Hoek.....	9913	10.29	12.87	1.41	147do.....	767	378	311	White Elk Vineyards.
9	California Muscatel.....	9913	10.67	13.34	1.41	190do.....	767	272	396	Pleasant Valley Wine Company.
930	Catawba, Ohio.....	9930	9.91	12.40	2.22	154do.....	632	292	264	Do.
13	California Sonoma Hoek.....	9845	9.66	12.05	1.39	190	Trace.....	422	213	167	Do.
21	Virginia Hoek, 1879.....	9905	9.58	11.96	1.39	166do.....	636	378	206	Do.
3534	White Concord, 1875.....	9934	8.42	10.56	2.42	160	None.....	789	332	366	Do.
45	Catawba.....	9903	10.39	13.71	2.10	135	Trace.....	833	480	282	Do.

II.—DRY WHITE WINES.—Continued.

No.	Name.	Specific grav.	Alcohol by weight.	Alcohol by volume.	Total residue.	Total ash.	Glucose.	Total acid, as tartaric.	Fixed acid, as tartaric.	Volatile acid, as acetic.	Maker.
		Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	
130	Catawba.....	.9928	10.52	13.15	2.19	.140	..	.542	.470	.068	Pleasant Valley Wine Company.
48	Brocton Catawba.....	.9890	12.28	15.30	2.09	.121	Trace	.780	.385	.323	G. E. Ryckman.
50	California Riesling Hock.....	.9932	9.00	11.26	1.67	.223	Trace	.619	.211	.508	Dressel & Co.
68	California Hock.....	.9935	9.07	11.35	1.92	.208	do	.785	.242	.354	Do.
72	Catawba.....	.9938	7.75	9.70	1.34	.117	..	.598	.272	.205	Kelly Island Wine Co.
85	Jersica, 1863.....	.9919	9.64	12.05	1.91	.138	Trace	.726	.387	.351	Julius Hineke.
86	Catawba, 1868.....	.9944	7.16	8.36	1.44	.167	do	.800	.350	.280	Do.
93	Catawba, Missouri.....	.9911	8.88	11.08	1.67	.129	do	.772	.387	.308	Isaac Cook.
106	Dry Muscat.....	.9928	9.14	11.44	1.82	.150	do	.619	.248	.289	Dreyfus & Co.
107	White Zinfandel.....	.9911	9.02	11.26	1.47	.139	do	.590	.257	.290	Do.
108	Riesling.....	.9918	9.64	12.65	1.72	.221	do	.636	.210	.389	Do.
109	Gutedel.....	.9920	9.36	11.70	1.58	.196	do	.726	.212	.411	Do.
110	Hock.....	.9959	7.73	9.70	1.73	.209	do	.723	.211	.410	Do.
118	"A. A." Catawba, second quality.....	.9929	7.69	9.62	1.29	.192	None	.710	.257	.362	Wehrle, Werk & Co.
119	"A. A." Catawba, first quality.....	.9912	9.09	11.35	1.28	.164	Trace	.672	.309	.371	Do.
123	Delaware.....	.9940	7.03	8.80	1.51	.255	None	.664	.256	.350	Do.
125	"Mount Vernon".....	.9962	7.73	9.70	2.07	.202	Trace	.669	.286	.306	C. A. Heineken.
128	Natue wine, N. Mex., 1877.....	.9894	10.55	13.15	1.80	.275	Trace	.485	.121	.291	L. & H. Huning.
138	Sonoma Mission, 1878.....	.9932	8.44	10.56	1.63	.184	do	.619	.311	.246	Gretsch & Mayer.
140	Sonoma Riesling, 1877 (?).....	.9926	10.53	13.15	2.28	.223	do	.695	.332	.290	Do.
141	Sonoma Riesling, 1879.....	.9906	10.54	13.15	1.70	.194	do	.575	.257	.254	Do.
142	Sonoma Mission, 1879.....	.9935	8.30	10.38	1.67	.193	do	.619	.317	.242	Do.
143	Sonoma Gutedel, 1879.....	.9921	9.50	11.87	1.71	.197	do	.589	.287	.242	Do.
144	Dry Muscat, 1874 (?).....	.9921	9.92	12.40	1.66	.256	do	.816	.302	.411	Do.
145	White Zinfandel, 1878.....	.9928	9.56	11.96	1.96	.161	do	.761	.393	.324	Do.
146	White Zinfandel, 1879.....	.9927	8.80	11.00	1.87	.182	do	.740	.332	.326	Do.
148	Ohio Catawba, 1879.....	.9892	10.25	12.77	1.63	.113	do	.728	.424	.243	Do.
149	Ohio Catawba, 1880.....	.9935	9.28	11.61	1.80	.111	do	.698	.478	.128	Do.
150	Hellena (?) 1879.....	.9954	7.80	9.78	2.06	.229	do	.693	.271	.250	Do.
156	Riesling, 1880.....	.9856	8.08	10.38	2.06	.355	None	.603	.319	.203	Poeschel, Scherer & Co.
159	Rulander, 1880.....	.9914	10.46	13.65	1.90	.199	Trace	.545	.302	.194	Do.
161	Delaware, 1880.....	.9932	9.35	11.70	1.88	.255	do	.502	.332	.184	Do.
165	Taylor, 1880.....	.9921	10.37	12.96	1.99	.185	do	.732	.317	.332	Do.
166	Goethe, 1880.....	.9962	8.55	10.73	1.85	.223	do	.693	.301	.314	Do.
173	Catawba, 1879.....	.9941	7.55	9.45	1.57	.114	None	.830	.468	.290	H. T. Dewey & Son.
175	Iona and Catawba, 1871.....	.9906	9.73	12.13	1.91	.103	Trace	.742	.469	.218	Do.
176	Iona, 1870.....	.9892	12.05	15.02	1.62	.090	do	.810	.561	.199	Do.

III.—SWEET WINES.

178	Catawba, 1880.....	.9937	7.62	9.54	1.39	.148	do.....	.755	.317	.350	Fritz Baier.
179	White Concord, 1880.....	1.0105	8.02	10.21	1.34	.183	do.....	.481	.252	.183	Do.
163	Herbemont, 1880.....	.9928	7.78	9.80	1.60	.146	None.....	.562	.302	.508	Poeschel, Scherer & Co.
164	Catawba, 1880.....	.9934	8.64	10.82	1.63	.178	Trace.....	.574	.362	.170	Do.
134	White Concord, 1875.....	1.0022	7.03	8.88	3.66	.158	1.20.....	.784	.404	.364	White Elk Vineyards.

28	Port, Iowa.....	1.0116	10.25	13.05	6.89	0.178	4.15	0.697	0.430	0.214	White Elk Vineyards.
40	Port, New York.....	1.0207	13.77	17.70	9.83	.142	7.77	.808	.548	.208	Pleasant Valley Wine Company.
41	California Port.....	1.0258	16.93	21.80	11.43	.467	8.00	.790	.307	.386	G. E. Ryckman.
46	Boston Port, New York.....	1.0508	16.10	13.24	17.04	.139	11.80	.828	.600	.182	Kohler & Froling.
52	California Port.....	1.0297	10.10	20.89	12.37	.285	5.78	.510	.320	.152	Alfred Speer.
57	Speer's Port, New Jersey.....	1.0213	13.67	17.59	10.69	.369	7.44	.705	.347	.286	Dreyfus & Co.
70	California Port.....	1.0139	14.78	18.88	8.55	.355	4.49	.755	.320	.348	Do.
111	do.....	1.0122	15.58	19.87	8.36	.347	5.88	.870	.196	.139	Perkins, Stern & Co.
103	do.....	1.0228	12.03	10.33	10.33	.300	8.60	.486	.238	.198	Do.
10	"Sunny Slope," California.....	1.0405	11.53	15.12	12.96	.336	11.57	.433	.259	.138	Pleasant Valley Wine Company.
129	Port, New York.....	1.0296	11.44	14.84	11.45	.190	9.18	.693	Gretsch & Mayer.
153	Los Angeles, California.....	1.0339	12.68	16.52	14.18	.345	11.39	.508	.348	.128	

39	California Sherry.....	.9873	14.42	17.92	1.95	.197	.61	.532	.231	.241	Urbana Wine Company.
2	Sherry, New York.....	1.0074	13.87	17.59	6.83	.166	4.81	.689	.209	.323	Perkins, Stern & Co.
53	Sherry.....	.9944	20.69	25.17	5.17	.479	2.97	.694	.332	.290	Do.
105	Marsala.....	1.0052	16.06	20.33	6.42	.428	3.53	.626	.416	.166	Dreyfus & Co.
69	California Sherry.....	.9987	12.84	16.15	4.70	.202	4.70	.721	.248	.380	Do.
113	do.....	.9942	13.42	16.80	3.91	.198	2.20	.573	.232	.273	Alfred Speer.
58	Speer's Sherry.....	.9949	17.62	22.09	4.89	.219	3.33	.476	.271	.164	

14	"Dry Sillery".....	1.0293	9.22	11.96	10.70	.104	7.34	.685	.438	.198	Henriot & Co. (?)
24	"Great Western," extra dry.....	1.0268	9.05	11.10	10.41	.131	9.08	.818	.365	.362	Pleasant Valley Wine Company.
132	do.....	1.0285	8.35	10.82	11.07	.130	8.79	.501	.394	.186	Do.
23	"Grand Prize," medium dry.....	1.0228	9.75	12.49	9.15	.134	8.21	.821	.323	.398	Arpad Haraszthy.
26	"Eclipse," extra dry.....	1.0174	9.26	11.87	7.78	.149	6.51	.885	.295	.472	Do.
27	"Gold Seal".....	1.0402	8.26	10.82	13.31	.110	12.02	.880	.447	.346	Urbana Wine Company.
35	"Saus Pareil".....	1.0272	5.78	7.48	9.00	.147	8.74	.862	.438	.339	William H. Mills.
167	do.....	1.0308	8.07	10.47	10.30	.153	8.78	.825	.626	.159	Do.
168	La Diamant.....	1.0217	8.40	10.82	8.73	.138	7.54	.564	.411	.122	Do.
169	Norton's Virginia (red), 1872.....	1.0188	6.24	8.01	8.58	.164	7.24	.692	.515	.142	Do.
88	Cook's Imperial.....	1.0207	8.41	10.82	8.47	.130	7.23	.779	.470	.247	Isaac Cook.
101	do.....	1.0222	7.03	9.04	7.80	.114	7.02	.851	.411	.552	Do.
102	"Red Cross" (red).....	1.0264	10.02	12.96	11.23	10.11	.570	.322	.198	M. Wark & Son.
183	do.....	1.0265	8.58	11.08	11.01	.102	9.01	.567	.386	.145	Do.
174	Catawba, 1878.....	1.0233	7.64	9.86	8.57	.114	6.60	.572	.423	.119	H. T. Dewey & Son.

Analyses of American wines, &c.—Continued.

III.—SWEET WINES—Continued.

No	Name.	Specific grav- ity.	Alcohol, by weight.	Alcohol, by volume.	Total residue.	Total ash.	Glucose.	Total acid, as tartaric.	Fixed acid, as tartaric.	Volatile acid, as acetic.	Maker.
		Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	
<i>Sweet Catawbas.</i>											
73	Pass Island.....	1.0338	11.68	15.21	14.49	.152	11.00	.595	.296	.239	White Elk Vineyards.
30	Iowa, 1871.....	1.0101	9.89	12.58	7.23	.211	4.01	.668	.318	.280	Pleasant Valley Wine Company.
44	New York.....	1.0219	12.98	16.70	11.13	.120	8.98	.519	.382	.110	Do.
131do.....	1.0231	13.40	17.26	10.78	.140	8.87	.367	.289	.063	Do.
184do.....	1.0199	15.40	19.78	11.42	.126	9.49	.560	.406	.123	G. E. Ryckman.
47	Brocton, New York.....	1.0512	10.71	14.18	16.71	.113	15.22	.714	.471	.194	
<i>Miscellaneous.</i>											
54	Sweet Muscatel.....	1.1022	13.51	18.58	31.34	.371	25.37	.753	.421	.266	Perkins, Stern & Co.
71do.....	1.0245	17.33	22.36	11.70	.218	11.59	.366	.234	.106	Dreyfus & Co.
117do.....	1.0437	17.08	22.46	17.09	.126	16.94	.331	.273	.046	Do.
151	Los Angeles Muscatel.....	1.0418	12.81	17.08	13.61	.553	13.44	.173	.360	.138	Gretsch & Mayer.
42do.....	1.0440	8.96	11.79	14.41	.196	12.48	.489	.310	.143	
104do.....	1.0381	10.63	13.90	13.63	.249	13.25	.347	.254	.074	Perkins, Stern & Co.
116do.....	1.0465	13.77	18.14	15.94	.230	14.81	.430	.315	.092	Dreyfus & Co.
152	Los Angeles Angelica.....	1.0493	14.77	18.78	18.04	.177	15.90	.466	.314	.122	Gretsch & Mayer.
49	Brocton Sweet Regina.....	1.0515	9.71	12.87	16.52	.101	15.31	.628	.465	.130	G. E. Ryckman.
51	Gerke's White Wine.....	1.0023	11.67	14.74	5.42	.341	2.21	.673	.245	.342	Henry Gerke.
95	Sweet Delaware, 1879.....	1.0320	8.73	11.35	12.07	.118	10.27	.799	.355	.355	W. J. Green.
96	Sweet Concord, 1880.....	1.0224	8.46	16.91	8.41	.132	7.12	.601	.358	.194	Do.
98	Scuppernong, 1880.....	1.0100	8.50	10.82	5.71	.111	1.78	.653	.252	.321	Do.
99	Scuppernong, sweet, 1878.....	1.0404	9.06	11.87	14.13	.132	11.56	.758	.823	.348	Do.
100	Scuppernong, dry, 1879.....	.9948	10.72	13.43	3.39	.108	1.31	.925	.346	.463	Do.
94	Norton's Virginia "Claret," 1880.....	.9962	12.27	15.40	3.61	.126	1.31	.828	.295	.426	Do.
43	California Malaga.....	1.0239	13.72	17.70	11.00	.423	8.59	.659	.264	.316	Henry Gerke.
177	Delaware, 1875.....	1.0111	7.74	9.86	6.45	.260	3.36	.593	.430	.130	H. T. Dewey & Son.
<i>Brandies.</i>											
23	Pure Grape.....	.9272	46.00	53.70	.125111	Monticello Wine Company.
112do.....	.9241	43.66	51.38	B. Dreyfus & Co.
170	Brandy, 1876.....	.9399	43.81	51.58	Trace.....075	H. T. Dewey & Son.

Averages and extremes of American dry wines.

Constituents, &c.	Dry red wines.			Dry white wines.		
	Average (64 analyses).	Highest.	Lowest.	Average (51 analyses).	Highest.	Lowest.
Specific gravity.....	.9933	1.0011	.9894	.9926	1.0105	.9845
Alcohol, by weight..... per cent.	8.92	12.21	5.71	9.35	13.94	7.03
Alcohol, by volume..... do.	11.04	15.21	7.17	11.70	17.37	8.80
Total residue..... do.	2.28	3.16	1.65	1.75	2.64	1.18
Total ash..... do.	0.231	0.532	0.130	0.181	0.335	0.090
Glucose..... do.	Traces.	0.450	None.	Traces.	0.300	None.
Total acid, as tartaric..... do.	0.723	0.997	0.511	0.680	0.855	0.422
Fixed acid, as tartaric..... do.	0.360	0.646	0.226	0.313	0.561	0.121
Volatile acid, as acetic..... do.	0.290	0.517	0.138	0.294	0.508	0.068

A very excellent monograph on California wines has been very recently published by Dr. George Baumert,¹ of the University at Halle. Although the analyses were made on only eight samples, the examination was very complete, including a search for adulterations; then follows a very exhaustive discussion of the composition, together with a comparison of the samples analyzed, and similar wines made in Germany, so that the paper is a very interesting and instructive contribution to the literature of American wines. I shall have occasion to allude to it again in discussing the subject of adulterations. Other analyses of American wines published in German periodicals and alluded to by Baumert are: "Investigation of a California Wine" by A. Kayser;² "Two Analyses of California Wines, Riesling and Zinfandel," by A. Stutzer;³ "Analysis of Two California Wines, Zinfandel and Gutedel Cabinet," by J. L. de Fremery.⁴

METHODS OF ANALYSIS.

The methods of analysis used in the present investigation of wines can be conveniently arranged under two heads: First, those designed for the establishment of the composition of the sample examined; and, second, those employed in the search for adulterants.

The determinations embraced under the first head are: Specific gravity, alcoholic content, total solids, acidity, content of sugars, of glycerine, of tannic acid, and of ash. These are the principal and most important determinations in a wine analysis, though there are several others which are sometimes made, such as the nitrogenous content, phosphoric acid, chlorine, malic, succinic, and citric acids, &c.

In my investigation of methods for wine analysis I have drawn especially upon three sources: First, the methods in use in the Municipal Laboratory in Paris, and set forth in the report already quoted under malt liquors; second, the methods officially authorized in the health

¹ Landw. Versuchs-Stationen, 1886, 33, 39.

² Mitth. d. Bayr. Gewerbe-Museum zu Nürnberg, 1879, Nr. 19.

³ Rep. der anal. Chem. 1882, Nr. 14.

⁴ Ber. der Deutschen Chem. Gesell., 1885, 426.

offices in Germany, decided upon by the "Berlin Commission;" third, the methods adopted by the Bavarian chemists.

It would seem that in France and Germany, where wine analysis has been carried on for so many years, and to so large an extent, the methods in use would be as well defined and exact as is possible in such work; nevertheless, none of the methods have been accepted without a thorough trial of their accuracy, unless otherwise indicated.

The estimation of several of the constituents of wine is so similar to the same determination in beer, that it will be necessary simply to refer to the part on beer analysis, thus avoiding unnecessary repetition.

SPECIFIC GRAVITY.

This is taken with the pycnometer, in the same manner as with beer.

ALCOHOL.

The estimation of the alcohol can be made in the same manner as in beer, with a few precautions. Thus, in the distillation method it is better to neutralize the sample taken with carbonate of soda, or standard alkali, and a slight addition of tannin will generally be found necessary to prevent frothing. If the indirect method is used, more accurate results will be obtained by neutralization of the sample taken by shaking up with magnesia before the specific gravity is taken. In the present work I have made use of the distillation method exclusively, weighing out the sample taken, about 50 grams, making up to about 100cc., and distilling off 50cc., which is also weighed. Tables for the calculation of alcohol content from the specific gravity have already been given page 285).

EXTRACT OR TOTAL SOLIDS.

The determination of the extract in wine presents the same difficulties as with beer on account of the content of glycerine, which is greater in the former than the latter. For this reason the indirect method would seem especially applicable, but the difficulty here is that there is a larger amount of volatile acid present, which is only partially driven off with the alcohol, and the solution density of the solids of wine have not been so well established as that of malt extract, so that different tables and factors vary widely.¹

For the direct estimation the French method is to evaporate 25cc. in a flat-bottomed dish, with vertical sides, and dry the residue to a constant weight in a water bath. The Berlin method is as follows:

EXTRACT.

Fifty cubic centimeters of wine are measured (at 15° C.) into a platinum dish (85mm. in diameter, 20mm. in height, and of 75cc. capacity, weight about 20 grams), evaporated on the water bath, and the residue heated 2½ hours at 100° C. Of wines rich in sugar, *i. e.*, containing over 5 grams sugar in 100cc., a smaller amount should be taken, so that the weight of extract shall not exceed 1, or at most 1.5 grams.

¹ Baumert found in his work on California wines that the tables of Schulze (Zeit Anal. Chem., 1880, 104) intended for beer extracts gave results in the indirect estimation which approximated much more closely to the results by direct estimation than figures obtained from Hager's tables (Chem. Cent., 1878, 415), which were intended especially for wine extract.

The Bavarian Chemists' Union depart from this method by drying the residue to a constant weight.

It is of great importance that chemists should adopt a uniform method of analysis, as results differ considerably by different methods. This is shown by the following results obtained from the samples analyzed by me, most of which were subjected to analysis by both methods, that is, drying to a constant weight and drying a certain length of time. In the very sweet wines, of course, such as the Angelica, Muscat, &c., containing so large a percentage of sugar, it can only be satisfactorily determined by using a small quantity, allowing it to flow well over the bottom of the dish, and drying to a constant weight as nearly as possible.

Comparison of methods for the estimation of extract in wine.

Serial Number.	Drying for 2½ hours at 100° C.	Drying to a constant weight.	Serial Number.	Drying for 2½ hours at 100° C.	Drying to a constant weight.
4995	2.69	2.00	5089	2.01	1.22
4996	2.82	2.26	5094	2.30	1.52
4997	2.08	1.44	5095	2.96	2.18
4998	1.57	1.16	5096	2.92	1.71
4999	2.18	1.75	5097	1.82	1.18
5000	2.24	1.74	5098	1.90	1.34
5001	4.13	3.82	5099	2.20	1.43
5002	9.78	9.30	5100	2.47	1.96
5004	9.62	9.53	5101	2.40	1.82
5005	2.51	2.09	5103	2.19	1.71
5081	1.77	1.16	5104	2.42	1.82
5083	1.80	1.16			
5084	2.12	1.39	average	2.98	2.38
5088	3.64	2.83			

I have adopted the results obtained from the estimation by drying to a constant weight, and would recommend the following procedure for the determination of extract in wine. Weigh out 10 to 50 grams of the wine (according to its content of sugar) into a flat-bottomed platinum dish, evaporate on the water bath and dry the residue at 100° C. in an air bath, until a constant weight is obtained. This may be known to be the case when by weighing the dish and contents at frequent intervals (15 minutes) the losses in weight are found to be slight and equal for the same length of time of drying.

ACIDITY.

The total acidity of wines may be determined by titration with $\frac{N}{5}$ or $\frac{N}{10}$ alkali. The end reaction may be observed by means of a drop brought into contact with delicate litmus paper, which, while not entirely satisfactory, still is probably the best method we have at hand, any means for decolorizing the wine so as to admit of the use of other indicators being likely to change the content of acid. I have found that by the use of turmeric paper in connection with the litmus, the establishment of the point of neutralization is greatly facilitated,

Very white wines, if entirely free from carbonic acid, may be titrated with phenol-phthaline as an indicator. From 10 to 25cc. of wine may conveniently be taken for titration. It is very desirable to estimate both the fixed and volatile free acids in wine, and to this end the latter should be determined directly; all methods for this estimation by difference by evaporating the wine to dryness, and titrating the residue, having been shown to be faulty. The German method is to distill off the acetic and other volatile acids in a current of steam; and I have found it a most satisfactory one, giving very coincident results when carefully carried out, the only objection being that it is a little tedious. The theory of the method is that acetic acid, which forms the greater part of the pure volatile acids, while it does not distill off at a temperature lower than 120°C ., can still be distilled off at a lower temperature than its boiling point by passing through the liquid in which it is contained a current of steam, providing that the quantity of water passed through in the shape of steam is about four times as great as the quantity of the liquid in which the acid is contained. The manner of applying it is as follows:

Fifty cc. of the wine are measured into a flask of about 300cc. capacity, which is connected with a condenser by one tube which passes just below the rubber stopper, which is also perforated by another tube which reaches to the bottom of the flask and terminates there in a finely drawn out point; this tube is bent at right angles where it passes out of the flask, and connects it with a larger flask placed alongside which contains about 300cc. of water, being also provided with a safety tube. When the operation is begun, lamps are placed under the two flasks and the contents of both brought to a boil, when the flame under the flask containing the wine is lowered somewhat and the distillation so conducted that after 200cc. have distilled off the wine shall not be decreased to less than one-third or one-fourth of its original volume. The 200cc. of distillate is received in a properly graduated flask, and titrated with standard alkali, using phenol-phthaline as an indicator.

The number of cubic centimeters of normal alkali required for the titration of the volatile acids, subtracted from the number of cubic centimeters required for the neutralization of 50cc. of the original wine, gives the amount of alkali neutralized by the free *fixed* acids; the total free acids and free fixed acids are generally calculated as tartaric, the volatile as acetic acid.¹

BITARTRATE OF POTASH.

The German Commission gives two methods, as follows:

In two stoppered flasks two samples of 20cc. of wine each are treated with 200cc. ether-alcohol (equal volumes), after adding to one flask 2-3 drops of a 20 per cent. solution of acetate of potash. The mixtures are well shaken and allowed to stand 16 to 18 hours at a low temperature ($0-10^{\circ}\text{C}$.), the precipitate filtered off, washed with

¹ One cubic centimeter of normal alkali neutralizes .075 gram of tartaric, .06 gram of acetic acid.

ether-alcohol, and titrated. (The solution of acetate of potash must be neutral or acid. The addition of too much acetate of potash may cause the retention of some bitartrate in solution.) It is best on the score of safety to add to the filtrate from the estimation of the total tartaric acid a further portion of 2 drops of acetate of potash to see if a further precipitation takes place.

In special cases the following method is recommended for a control over the other:

Fifty cc. of wine are evaporated to the consistency of a thin sirup (best with the addition of sand), the residue brought into a flask by means of small washings of 96 per cent. alcohol, and with continual shaking more alcohol is gradually added, until the entire quantity of alcohol is about 100cc. The flask and contents are corked and allowed to stand 4 hours in a cool place, then filtered, and the precipitate washed with 96 per cent. alcohol; the filter paper, together with the partly flocculent, partly crystalline precipitate, is returned to the flask, treated with 30cc. warm water, titrated after cooling, and the acidity reckoned as bitartrate. The result is sometimes too high, if pectinous bodies separate out in small lumps, inclosing a small portion of free acids (this error may, however, be avoided by the addition of sand and thorough shaking). In the alcoholic filtrate the alcohol is evaporated, 0.5cc. of a 20 per cent. potassic acetate solution added, which has been acidified by a slight excess of acetic acid, and thus the formation of bitartrate from the free tartaric acid in the wine facilitated. The whole is now, like the first residue of evaporation, treated with (sand and) 96 per cent. alcohol, and carefully brought into a flask, the volume of alcohol increased to 100cc., well shaken, corked, allowed to stand in a cold place 4 hours, filtered, the precipitate washed, dissolved in warm water, and titrated, and for 1 equivalent of alkali 2 equivalents of tartaric acid are reckoned. This method for the estimation of the free tartaric acid has the advantage over the former of being free from all errors of estimation by difference.

I have preferred making the qualitative test for free tartaric acid separately from the bitartrate estimation, and for the latter I have used essentially the first method given, omitting, of course, the parallel precipitation with the addition of acetate of potash, modifying it by using only 80cc. of the alcohol-ether solution for the precipitation and allowing it to stand over night. The titration is nicely performed with phenolphthaline and decinormal soda with white wines; 1cc. decinormal soda corresponding to .0188 grams of potassic bitartrate. In the case of dark colored wines I passed them first through a small amount of bone-black, afterwards washing the bone-black thoroughly, so as to avoid the presence of coloring matter in the tartar precipitate.

SACCHARINE MATTER.

For the estimation of the saccharine matter use was made of the Department method of employing Fehling's solution, already referred to under malt liquors. The Germans usually employ the gravimetric estimation, with Soxhlet's modifications, but I believe the other to be fully as accurate, and much more convenient. The wine should be evaporated about one-third to remove the alcohol, and carbonate of soda added to neutralize the acid. In the case of dark colored wines it is necessary to decolorize and clarify them by the use of subacetate of lead or bone-black. If much excess of lead is used it should be removed with sulphate of soda, and if bone-black is used the first portions filtered should be rejected. The amount of reduction is calculated as dextrose.

ASH.

The ash may be estimated in the residue from 50 to 100cc. of the wine. If 50cc. have been used for the estimation of the extract, the same sample will serve conveniently for incineration. This operation is best performed in a muffle, and must be very carefully carried out, at as low a heat as possible. If the ash does not readily burn white, it should be treated with a little water to dissolve the alkali salts, the dish placed in such a position as to bring the water away from the undissolved ash, the water evaporated, and the incineration completed. Little difficulty is generally experienced in getting in this way a very satisfactory white ash.

GLYCERINE.

The estimation of glycerine in wines, if it could be made with exactness, would be a very important one, as the glycerine is produced by the fermentation of the sugar, and the quantity formed is presumably fairly constant for the same amount of sugar fermented. This being the case, the quantity of glycerine in a wine should be a good index of the quantity of sugar which had undergone fermentation, and would thus show whether alcohol had been added to the wine. Unfortunately, the amount of glycerine present is so small, and its exact estimation so difficult on account of its volatile nature, that it is rather an unsafe reliance. The Germans attach considerable weight to the determination in establishing the character of a wine, using the following method:

One hundred cc. of wine (sweet wines excepted) are evaporated in a shallow, porcelain dish, to about 10cc., a little sand added, and milk of lime to a strong alkaline reaction, and the whole brought nearly to dryness. The residue is extracted with 50cc. of 96 per cent. alcohol on the water bath with continual stirring. The solution is poured off through a filter and the residue exhausted by treatment with small portions of alcohol. For this 50 to 150cc. are generally sufficient, so that the entire filtrate measures 100 to 200cc. The alcoholic solution is evaporated on the water bath to a sirupy consistence. (The principal part of the alcohol may be distilled off, if desired.) The residue is taken up by 10cc. of absolute alcohol, mixed in a stoppered flask with 15cc. of ether and allowed to stand until clear, when the clear liquid is poured off into a glass-stoppered weighing glass, filtering the last portions of the solution. The solution is then evaporated in the weighing glass until the residue no longer flows readily, after which it is dried one hour longer in a water jacket. After cooling, it is weighed.

In the case of sweet wines (containing over 5 grams of sugar in 100cc.), 50cc. are taken in a good-sized flask, some sand added and a sufficient quantity of powdered slacklime, and heated with frequent shaking in the water bath. After cooling, 100cc. of 96 per cent. alcohol are added, the precipitate which forms allowed to separate, the solution filtered, and the residue washed with alcohol of the same strength. The alcoholic solution is evaporated and the residue treated as above.

In regard to the performance of the official method, as given above, Dr. Barth¹ adds the following commentaries and cautions:

In case the residue from the first evaporation with lime becomes entirely dry it should be moistened with a little alcohol, the residue removed from the sides of the

¹ Die Weinanalyse, p. 17.

dish with a spatula, and the entire mass rubbed up with a pestle to a uniform pasty mass, the pestle and spatula being rinsed with a little alcohol; in heating up the alcoholic paste with lime, bumping and spurting may be avoided by careful stirring; the heating and subsequent washing out with hot alcohol is necessary, however, to dissolve out the glycerine properly. In evaporating with both the alcoholic and the ether-alcohol solution, all violent boiling of the liquid must be avoided, or mechanical losses will occur. The best way is to place the vessels containing the solutions inside of beakers filled with water on the bath. The clearing up of the ether-alcohol solution can be hastened by energetic shaking in the stoppered flask containing it. The vessel in which the ether-purified glycerine is finally weighed should have vertical walls at least 40mm. in height. The losses which are caused by the volatility of glycerine cannot be entirely avoided, but may be reduced to a minimum by a careful observance of all the directions, even those which are apparently unimportant. That the loss of glycerine is smaller by heating in a drying oven than on the open water bath has been noticed in the estimation of the extract; the choice of weighing tubes also with proportionally high, vertical walls has for its object the lessening of the possibility of losses in weight.

For the estimation of the glycerine in sweet wines the following precautions should be observed: Sufficient powdered lime must be added to the wine to convert the whole of the sugar into its lime compound. The formation of the latter takes place gradually during the heating on the water bath. The mass becomes at first dark brown (special care is necessary to prevent its foaming over the neck of the flask), but when the saturation with lime is complete it becomes somewhat clearer, and, together with the characteristic smell of the sugar-lime, a caustic odor becomes manifest.

If the residue obtained from the concentration of the alcoholic solution remains somewhat thin even after cooling, it is not necessary to repeat the treatment with lime. The purification with ether-alcohol in the way described will be all that is necessary.

The above described method for glycerine estimation is intended to obtain the glycerine in a state of purity by its separation from all the other constituents of wine, either by their volatility, by their insolubility in alcohol, or their lime combinations, or finally by their insolubility in a mixture of one volume of alcohol with $1\frac{1}{2}$ volumes of ether. If pointed crystals appear on cooling, the presence of mannite is indicated. Since the separation of glycerine in an insoluble condition in a form or union peculiar to itself has not yet been accomplished, the extraction method must serve for its estimation, but the latter is only useful for the conclusions which are drawn from its results, when it is carried out with a strict observance of the preceding conditions.

Several methods have lately been proposed for the estimation of glycerine, and it was with the hope of some of them proving more exact and less tedious than the above that a somewhat hasty examination of these methods has been made.

Benedikt and Zsigmondy¹ published in 1885 a method for the estimation of glycerine by its oxidation to oxalic acid by permanganate of potash, precipitating the oxalic acid with calcium acetate, and determining it volumetrically by titration with acid. This method is also claimed by Fox and Wanklyn.² At the time of the publication of this method I made several trials of it on pure glycerine with very satisfactory results, and Allen³ has confirmed the accuracy attributed to it by

¹ Chem. Ztg. 9, 975; Analyst 10, 206.

² Chem. News 53, 15.

³ Analyst 41, 52.

Benedikt and Zsigmondy, and used it on saponified fats. It has never been applied to wine or beer, so far as I know.

Legler¹ has formulated a method, intended, as the author says in his paper, to supply the place of the method of the Berlin committee, and atone for its deficiencies. It depends on the oxidation of glycerine to carbonic acid by means of sulphuric acid and potassic bichromate.² The estimation of organic bodies by the oxidation of their contained carbon has been proposed and carried out by Cross and Bevan³ who operate in a dry, and by Burghardt,⁴ who operates in a wet, way. The operation was performed by Legler in a Will's carbonic acid apparatus, as follows:

The air flask contains the glycerine mixed with a saturated solution of potassic dichromate; the other contains, as usual, strong sulphuric acid. After the apparatus has been weighed, a little air is drawn out which causes some of the acid to mix with the chromate. A regular evolution of carbonic acid soon sets in, but must be assisted towards the last by gently boiling. The flask containing the sulphuric acid must be kept cool. When no more gas bubbles are formed, the apparatus is cooled by partial immersion in cold water, and the remaining carbonic acid is expelled by a current of dry air. The apparatus is now reweighed and the loss represents carbonic acid. The following equation shows the action taking place:



One part of glycerine therefore requires about 7.5 parts of $K_2Cr_2O_7$ and 10 parts of H_2SO_4 , but an excess of each is of course used.

The mode of procedure in operating on wine is as follows: The crude glycerine obtained from 100cc. of wine, after evaporation with 3cc. milk of lime and 2 grams of quartz, and extracting the mass with alcohol of 96 per cent., is, after weighing, diluted up to a definite bulk, and aliquot parts are taken for the ash and the oxidation process. A white wine, containing 8.54 per cent. alcohol and 2.07 per cent. solid matter, gave in 100cc. 1.4 grams crude glycerine, with .1278 grams ash, 25cc. of the glycerine diluted up to 50cc. yielding .725 gm. CO_2 = 1.10 per cent. glycerine. A duplicate experiment gave 1.47 crude glycerine with .136 ash, .710 CO_2 = .99 per cent. glycerine, the average thus being 1 per cent.⁵ and the relation between alcohol and glycerine as 100 : 11.7.

Estimation of glycerine in wine after it has been purposely added.

Three lots of 100cc. each of the same wine were mixed respectively with .125, .250, and .500 grams of glycerine, and analyzed as before. The results were as follows:

Crude glycerine.	Ash.	CO_2 (25cc. from 50cc.)	Glycerine in 100cc.
1.55	.1496	.80	1.115
1.75	.1400	.90	1.254
2.07	.1172	1.07	1.492

Allowing for the 1. per cent. of natural glycerine in the sample we obtain .115, .254, and .492 per cent. of glycerine.

¹ Rep. Anal. Chem. 6, 631; Analyst 12, 14.

² Chem. News 53, 297; also 55, 2.

³ Chem. News 55, 34; see also 55, 46.

⁴ *Ibid.*

⁵ The average is really 1.05 per cent., which would make some difference in the figures which follow.

The same wine was also submitted to an analysis by the old process, somewhat higher results being obtained, from which Legler concludes that his method gives lower but more correct figures. He also obtained promising results from beer and sweet wines. This method was tried on some of the samples analyzed in the Chemical Division, the analytical work being done by Mr. Felix Lengfeld.

The apparatus for the estimation of the carbonic acid was a specially designed one used for most carbonic acid estimates in the laboratory, and provided for the absorption of the dried gas by soda lime. A trial was first made with pure glycerine, which gave very satisfactory results, as follows: Weight of pure glycerine taken, .3645; weight glycerine obtained, .3605; difference, .004.

When it was applied to the wines, however, it was found that the results obtained varied widely from those obtained from the same samples by the old method, and instead of the variation being constant; it was found that sometimes the results were higher, sometimes lower, than by the old method, the manner of manipulation making a very marked difference in the quantities obtained. Thus when just about sufficient lime was taken to combine with the sugar present and the mass not evaporated very closely, higher results were obtained by Legler's process than by the old method, while if a large excess of lime was added and the whole evaporated very nearly to dryness the results were lower.

Thus Nos. 4998 and 4999, treated with considerable lime and evaporated nearly to dryness, gave the following results:

Numbers.	By Legler's method.	By old method.
4998.....	.326	.436
4999.....	.542	.797

While Nos. 4995, 5000, and 5002, treated with a smaller amount of lime, and not evaporated so closely, gave:

Numbers.	By Legler's method.	By old method.
4995.....	.972	.730
5000.....	1.123	.835
5002.....	1.549	.637

From these and from several other determinations, where the differences were still more marked, it was concluded that Legler's method as it stands now cannot be relied on to give constant results with wines, for though undoubtedly an accurate method of estimating pure glycerine, it is open to the same objection that applies to the method by oxidation with permanganate, viz, that other organic bodies are also oxidized, when present, and give too high results. In the extraction of the glycerine

from wine by alcohol other organic bodies are undoubtedly taken up by it (the most of which are removed in the old method, by the purification with ether-alcohol) which make the results too high. The only way that results could be obtained as low as the old method gave was as indicated above, by long treatment in the bath with a large excess of lime, and in these cases there was undoubtedly a loss of glycerine by evaporation, as the following experiments show :

1. .3645 grams pure glycerine were mixed with a small quantity of lime, alcohol and water added, the whole evaporated nearly to dryness on the water bath, extracted with alcohol, and the glycerine determined by Legler's method in the residue from the alcoholic solution ; result, glycerine .303, or a loss of .0615.

2. .3645 grams glycerine were taken and treated as above, except that a large amount of lime was added ; result, .275 ; loss, .0895.

3. .3645 grams were taken and treated as before, except that the evaporation was carried to dryness, the dish being allowed to stand on the bath about half an hour longer than in the other experiments ; result, .251 ; loss, .1135.

Hehner¹ has applied Legler's method to the estimation of glycerine in fats, with some modifications based upon the very important fact he claims to have established in his work, that "dilute solutions of glycerine (.6 glycerine to 1,000 of fluid) do not, as is commonly supposed, volatilize in concentrating the fluid, be it on the water-bath or over the naked flame."

Other methods that have been published by Benseman,² Amthor,³ and Medicus,⁴ are essentially slight modifications of the Berlin method, and can only be referred to here. Sulman and Berry,⁵ in their article on "The Examination of Commercial Glycerine," give a very thorough résumé of various methods in use up to the date of the article for the estimation of glycerine.

The most recent method published is by Diez,⁶ which I have not had time to investigate closely. The method is quite a novel one, and differs from the previously described methods in that it separates out the glycerine as an insoluble compound, viz, as a tribenzoate of glycerine. This is accomplished by shaking the (.5 to 1.0 per cent.) solution of glycerine with benzoyl chloride after an addition of alkali. As applied to dry wines it is described by the author as follows:⁷ "20cc. are evaporated to a moderately dry condition after the addition of lime. The residue is exhausted with 20cc. of hot, 96 per cent., alcohol. After cooling 30cc. of water-free ether are added, and filtered after standing, the filter being washed with water-free alcohol-ether (2:3). After the evaporation of the solvent the glycerine is dissolved in 10-20cc. of water,

¹ Analyst 12, 44.

² Chem. Zeit. 10, 554.

³ Rep. der. Anal. Chem., 1886, 12.

⁴ *Ibid*, 1886, 1.

⁵ Analyst 11, 12 and 34.

⁶ Zeit. f. Physio. Chem. 11, 472.

⁷ *Ibid.*, 480.

according to the quantity, and shaken up with 5cc. benzoyl chloride and 35cc. of 10 per cent. soda solution for 10–15 minutes without cessation and with frequent cooling. The *estergeme* or precipitate obtained, is collected upon a filter, washed and dried for 2 to 3 hours at 100° C.; 0.385 gram of the weighed precipitate corresponds to 0.1 gram glycerine." The objection to this process seems to be that it is not applicable directly to the wine or beer, but the glycerine must be separated out in a state of comparative purity before it can be converted into the benzoate, and there is still the liability to loss during the process of purification.

The results given in the table are by the old method.

TANNIN.

The estimation of tannin in wines, where considerable accuracy is required, may be made by the permanganate and indigo method, which has been so much discussed and modified in chemical literature. The following approximate method is given by the Berlin Commission:

In 10cc. of wine the free acids are, if necessary, reduced by the addition of standard alkali solution to .5 grams in 100cc. Then 1cc. of a 40 per cent. solution of acetate of soda is added, and finally, drop by drop, 10 per cent. solution of chloride of iron, avoiding an excess. One drop of the chloride of iron solution suffices for the precipitation of about .05 per cent. of tannin.

For the estimation of the bulk of the precipitate test-tubes are used, which are much narrowed at the bottom, with the constricted portion graduated into tenths of a centimeter. The following table gives the approximate content of tannin from the bulk of the precipitate after standing twenty-four hours:

Precipitate after 24 hours.	Tannin in the wine.	Precipitate after 24 hours.	Tannin in the wine.
cm.	Per cent.	cm.	Per cent.
0.1	0.003	1.0	0.033
0.2	0.007	2.0	0.066
0.3	0.010	3.0	0.10
0.4	0.013	4.0	0.13
0.5	0.017	5.0	0.17
0.6	0.020	6.0	0.20
0.7	0.023	9.0	0.30
0.8	0.027	12.0	0.40
0.9	0.030		

I can see no practical conclusions to be drawn from so indefinite a determination and have omitted it in my work, simply quoting it for the benefit of any one who might desire to make the estimation.

ANALYSES MADE BY THE DEPARTMENT OF AGRICULTURE.

In the work on wines during the present investigation, 70 samples, purchased in the market of Washington, were examined. Inasmuch as the analyses made in 1880 included so many samples, and represented very fairly the composition of the wine sold here, it was thought inadvisable to make a complete analysis of all the samples, especially as many of them were identical in origin with those examined by Mr. Parsons. Accordingly only about one-half the samples (36) were submitted to a very careful and complete analysis, the rest being examined for adulteration only, especially preservatives. Only those samples were chosen for complete analysis which did not correspond to any of the samples analyzed in 1880. The samples are all wines of American origin, of which by far the greater bulk of the wines consumed here consists. Most of the samples are Californian, a few coming from Virginia and other States. Several of the samples had foreign labels, in imitation of some imported wine of the same general class, but in each case the dealer admitted that the wines were American.

The time and scope allowed to the work did not admit of the extension of the investigation to imported wines.

Analyses of wines made by United States Department of Agriculture in 1887.

Designation.	Made in—	Vintage.	Serial number.	Number of analyses.	Specific gravity.	Alcohol by weight.	Alcohol by volume.	Extract.	Total acids as tartaric.	Fixed acids as tartaric.	Volatile acids as acetic.	Bitartrate of potash.	Reducing sugars as dextrose.	Glycerine.	Ash.	Polarization in degrees, cane-sugar scale.
<i>Red wines.</i>																
American Burgundy.....	California.....	1885	4964	1	.9903	11.93	14.74	Per et. 1.73	Per et. .390	Per et. .272	Per et. .067	Per et. .115	Per et. .390	Per et.	Per et. .176	0
Charbono.....	do.....	1885	4968	2	.9946	9.12	11.35	2.28	.498	.166	.267	.120	.324	.302	.324	1.9
Lenoir.....	do.....	1885	4969	3	.9951	10.43	12.96	2.25	.426	.277	.120	.120	.354	.354	.308	1.5
Burgundy.....	do.....	1884	4995	4	.9945	10.23	12.68	2.00	.870	.724	.121	.062	.093	.730	.255	1.0
Claret.....	do.....	1885	4996	5	.9943	10.61	13.15	2.26	.668	.535	.109	.076	.256	.588	.201	1.6
Zinfandel.....	do.....	1883	5005	6	.9945	9.87	12.22	2.09	.795	.688	.104	.057	.153	.893	.201	1.1
Burgundy.....	do.....	1885	5084	7	.9951	8.29	10.30	1.39	.383	.113	.216	.057	None.	.552	.286	0.2
St. Julien Claret.....	California.....	1885	5088	8	.9983	10.38	12.87	2.83	.728	.404	.211	.062	.508	.406	.280	0.
Claret.....	Virginia.....	5094	9	.9943	9.04	11.20	1.52	.680	.315	.124	.057	.124	.517	.294	1.
Zinfandel.....	California.....	5095	10	.9950	8.76	10.87	2.18	.765	.414	.281	.048	.250	.303	.342	1.
Claret.....	Virginia.....	5096	11	.9958	8.92	11.08	1.71	.525	.297	.183	.095	Trace.	.282	.396	0.2
Do.....	do.....	5099	12	.9949	8.43	10.47	1.96	.555	.279	.221	.086	.051	.421	.307	0.
Do.....	New Jersey.....	5100	13	.9947	9.94	12.31	1.96	.735	.450	.228	.029	.145	.308	.229	0.
Do.....	Virginia.....	5101	14	.9969	7.78	9.68	1.82	.705	.600	.084	.133	None.	.492	.252	0
Do.....	California.....	5103	15	.9923	10.45	12.95	1.71	.668	.443	.180	.029	None.	.370	.453	0.5
Do.....	do.....	5104	16	.9937	10.01	12.40	1.82	.585	.392	.154	.048	None.	.348	.360	0.7
Average.....9946	9.66	11.95	1.94	.611	.397	.169	.068	.104	.490	.290
<i>White wines.</i>																
Moselle.....	California.....	1884	4997	1	.9911	10.91	13.52	1.44	.735	.592	.118	.094	.073	.394	.247	2.2
Riesling, gray.....	do.....	4998	2	.9917	9.37	11.61	1.16	.750	.595	.128	.150	.081	.436	.202	2.4
Riesling, white Johannisburg.....	do.....	4999	3	.9919	10.91	13.52	1.75	.563	.451	.092	.059	.325	.797	.203	2.0
Sauterne.....	do.....	5000	4	.9882	13.35	16.52	1.74	.488	.385	.082	.062	.394	.685	.181	3.2
Dry Catawba.....	do.....	5081	5	.9913	10.11	12.49	1.16	.683	.448	.156	.198	.147	.370	.240	1.8
California Riesling.....	do.....	5083	6	.9914	9.95	12.31	1.16	.690	.471	.175	.189	.139	.427	.260	0.8
Riesling.....	California.....	5089	7	.9927	9.01	11.17	1.22	.668	.548	.096	.142	.109	.585	.203	0.5
Do.....	do.....	5097	8	.9920	9.64	11.96	1.18	.713	.530	.146	.255	.980	.540	.226	0.
Berger.....	do.....	5098	9	.9903	10.74	13.32	1.34	.698	.464	.187	.236	None.	.365	.222	0.4
Average.....9912	10.44	12.94	1.35	.665	.498	.131	.152	.250	.328	.220

Designation.	Made in—	Vintage.	Serial number.	Number of analyses.	Specific gravity.	Alcohol by weight.	Alcohol by volume.	Extract.	Total acids as tartaric.	Fixed acids as tartaric.	Volatile acids as acetic.	Bitartrate of potash.	Reducing sugars as dextrose.	Glycerine.	Ash.	Polarization in degrees, cane-sugar scale.
<i>Sweet wines.</i>																
Sherry.....	California..	1883	5001	1	.9929	16.16	Per ct. 19.87	Per ct. 3.82	Per ct. .638	Per ct. .445	Per ct. .157	Per ct. .039	Per ct. 1.850	Per ct. .606	Per ct. .312	12.
Do.....	do.....	5090	2	.9939	15.99	19.68	3.38	.510	.390	.096	.114	2.800	.278	.215	6.6
Port.....	do.....	5091	3	1.0432	15.39	18.93	15.38	.683	.431	.202	.076	8.928	.190	.602	29.2
Sweet Burgundy.....	do.....	1884	5002	4	1.0161	15.53	19.08	9.30	.615	.490	.092	.057	6.150	.657	.415	30.2
Sweet Catawba.....	do.....	1886	5087	5	1.0145	14.50	17.87	8.39	.518	.410	.087	.086	6.650	.113	.118	8.
Do.....	do.....	5102	6	1.0357	10.98	13.60	13.20	.463	.303	.130	.039417	.384	8.8
Tokay.....	California..	1884	5004	7	1.0167	14.58	17.92	9.53	.518	.384	.118	.039	6.110	.206	.262	30.2
Sweet Muscatel.....	do.....	1884	5003	8	1.0311	12.99	16.05	17.20	.375	.325	.025	.048	13.050	.192	.236	30.6
Muscatel.....	do.....	5092	9	1.0380	15.45	19.00	13.64	.563	.383	.144	.057	11.111	.102	.360	23.4
Angicola.....	do.....	1884	4994	10	1.0492	12.54	13.49	16.27	.373	.330	.030	.132	14.200	.140	.686	30.0
Do.....	do.....	5093	11	1.0433	15.37	18.90	13.24	.360	.285	.060	.039	11.873	.052	.249	29.9
Average.....	1.0261	14.50	17.85	11.21	.511	.378	.104	.067	8.48	.260	.351

Maximum, minimum, and mean composition of the samples examined.

Constituents.	Sixteen samples red wines.			Nine samples white wines.			Eleven samples sweet wines.		
	Maximum.	Minimum.	Mean.	Maximum.	Minimum.	Mean.	Maximum.	Minimum.	Mean.
Specific gravity9983	.9903	.9946	.9919	.9882	.9912	1.0511	.9929	1.0261
Alcohol by weight..... per cent..	11.93	7.78	9.66	13.35	9.01	10.44	16.16	10.98	14.50
Alcohol by volume..... do.....	14.74	9.68	11.95	16.52	11.17	12.94	19.87	13.60	17.85
Extract..... do.....	2.83	1.39	1.94	1.75	1.16	1.35	17.20	3.38	11.21
Total acids as tartaric..... do.....	.870	.383	.611	.750	.488	.665	.683	.360	.511
Fixed acids as tartaric..... do.....	.724	.113	.397	.595	.385	.498	.490	.285	.378
Volatile acids as acetic..... do.....	.281	.084	.169	.187	.082	.131	.202	.025	.104
Bitartrate of potash..... do.....	.133	.029	.068	.255	.039	.152	.132	.039	.067
Reducing sugars as dextrose..... do.....	.508	none.	.164	.980	none.	.250	15.05	1.85	8.48
Glycerine..... do.....	.893	.303	.490	.835	.365	.528	.657	.052	.260
Ash..... do.....	.453	.176	.290	.260	.181	.220	.686	.118	.351

A comparison of the composition of American wines with those of other countries, as shown in these analyses, and a discussion of the points of difference or agreement would be a most interesting and instructive task. It would require considerable space, however, for a proper presentation of the subject, and must be omitted in the present investigation, as not being of so direct importance to the question of the adulteration of wines.

THE ADULTERATION OF WINES.

The adulteration of wine has been practiced from a very early date in those countries where the consumption is large. It has increased in amount and in the skillfulness of its practitioners until at the present day it requires for its detection all the knowledge and resources which chemical science can bring to bear upon it, and even then a large part doubtless escapes detection. It must be remembered, however, that in Europe the definition of adulteration has rather a wide scope, including the addition of substances which are simply diluents. The Paris Laboratory considers as a fraud "the addition of any substance for the purpose of gain which changes the composition of the natural wine." In Germany, on account of the northern situation of the country, it is permitted to the wine-growers in bad years, when the grapes contain a relatively high percentage of acid and a low percentage of sugar, to make use of pure sugar as an addition to the must, which addition is not considered as an offense against the adulteration laws, so long as the product is sold as "wine" simply. The amount of water added with the sugar must not be greater than twice the weight of the former, and the product must not be offered for sale as "natural wine."

By far the greater part of the adulteration carried on in the European countries consists of this addition of water (*mouillage*) and sugar (*sucrage*). Such wines result from the methods of manufacture already

described—petiotization, gallization, and chaptalization. For the detection of such wines it is necessary to establish maximum and minimum limits for the principal constituents of wines, and the relation in which these constituents stand to one another. To establish these limits is rather difficult, and requires a large series of analyses extending over many years. The constituents most relied on for the establishment of the character of a wine in judging whether it has been diluted or not are: the extract, content of free acid, and the relation between the extract and mineral matters.

In Germany the lowest limit of the extract in a natural wine is placed at 1.5 grams in 100cc., and after subtracting the amount of free fixed acids calculated as tartaric from the extract, the amount of the latter left must be not less than 1.1 grams; or after the subtraction of the total free acids as tartaric, not less than 1. gram in 100cc. In the Paris Laboratory no exact limits are set, the decision being left to the judgment of the analyst, after a careful comparison of the sample with analyses of previous similar wines done in the laboratory in past years. The sugar added is often glucose, which introduces into the wine substances more or less injurious, depending upon its character. Ordinary glucose contains usually 10 or 15 per cent. of non-fermentable substances, which serves to increase the weight of the extract, thus masking the addition of water. Its fermentation gives rise to the formation of a small quantity of amylic alcohol, which increases the intoxicating effect of such wines, and causes headache and nausea in those partaking of them.

In the detection of this substance advantage is taken of the non-fermentable character of the dextrin it contains, and of the presence of amylin, a non-fermentable, highly dextrorotatory body found in commercial starch sugar. 50cc. of wine, after driving off the alcohol by evaporation, are subjected to fermentation by the addition of a little well-washed yeast. After the fermentation is complete the liquid is clarified by means of lead or bone black, and polarized. If starch sugar has been used a strong rotation to the right will be observed, while if the wine was natural, or if the sugar used was cane sugar, there would be no rotation. The following procedure is also given by the German Commission:

Two hundred and ten cubic centimeters of the wine are evaporated to a thin sirup on the water-bath after the addition of a few drops of a 20 per cent. solution of acetate of potash. To the residue is gradually added with continual stirring 200cc. of 90 per cent. alcohol. The alcoholic solution when perfectly clear is poured off or filtered into a flask and the alcohol driven off until only about 5cc. remain. The residue is treated with about 15cc. water and a little bone-black, filtered into a graduated cylinder and washed with water until the volume measures 30cc. If, now, this liquid shows a rotation of more than $+0.5$, Wild, the wine contains the unfermentable matters of commercial potato sugar (amylin).

I have made no trial of these methods on American wines, and give them simply as a reference. It is well known that American starch

sugar, made from corn, is quite different in composition from the European article, which is usually made from potato starch, and I do not know that the presence of amylin has been demonstrated in the American article. Whether the latter contains much dextrin or not depends upon the character of the "glucose" used; if it is the liquid glucose, I can testify from experience that it contains a considerable percentage of dextrin; but if it is the highly converted "grape sugar" or solid glucose that is used, probably not much dextrin is introduced into the wine from it.

Fraudulent wines are frequently made from raisins or dried grapes in France, and, according to French authorities, can easily be recognized by their high percentage of reducing sugar, and left-handed polarization after fermentation.

The *plastering* of wines, which is also very extensively carried on in France, consists in adding to the wine or must a large excess of gypsum, or sulphate of lime.

The sulphuric acid of the lime salt replaces the tartaric acid which is combined with potash, and forms an acid sulphate of potash, while the tartaric acid separates out as tartrate of lime. The operation is said to give the wine a brighter color and to enhance its keeping qualities, probably by a mechanical carrying down of some of the albuminous matters. Some authorities seem to regard the addition as a pardonable one on this account, but most condemn it. It certainly introduces into the wine a salt entirely foreign to the grape and of a more objectionable nature than that which it supplants, viz, the bitartrate of potash. Both Germany and France are in accord as to the limit of sulphuric acid which can be used in a wine, requiring a wine with a content of SO_3 , corresponding to over 2 grams of potassic sulphate (K_2SO_4) per liter, to be designated as a plastered wine. This figure affords a pretty wide margin, for the average content of wines, according to most observers, is not over one-fourth of the standard, or .5 grams K_2SO_4 to the liter.

The determination of the sulphuric acid can be made directly by precipitation of the wine with barium chloride, but is much more conveniently and rapidly carried out as follows:

Take 14 grams of pure, dry, crystallized barium chloride, together with 50cc. hydrochloric acid, and make up to a liter. If 10cc. of wine are used, every 1cc. of this solution required indicates a content of 1 gram K_2SO_4 to the liter of wine; accordingly to several portions of wine of 10cc. each are added, respectively, 0.7, 1, 1.5, 2cc. and more if necessary, the solution heated, and allowed to stand. When cool they are filtered, and a little more barium chloride added to each test. The appearance or non-appearance of a further precipitate in the different tests will show between what limits the content of SO_3 lies.

The use of starch sugar is also likely to introduce sulphates into the wine.

Fortification of wine consists in the addition of alcohol derived from some other source. The alcohol may be added either to the must or the

wine. It allows of better incorporation with the wine if it is added to the must before fermentation. In either case, however, it precipitates a part of the constituents originally dissolved, lowers the quantity of extract, deprives the wine of its original bouquet and flavor, and renders it more heady and intoxicating. The least objectionable addition is alcohol distilled from grapes; but the high price of the latter renders it much less likely to be used than corn spirit, which contains considerably more fusel oil. The practice of fortification prevails especially in the more southern wine-growing countries, as Portugal, Spain, and the South of France. Growers in those countries declare it to be a necessary addition in their warm climates for the preservation of the wines, as these latter contain a considerable quantity of unfermented sugar, which would soon produce the souring of the wine if the alcoholic content were not greater than can be obtained by fermentation. In France, for ordinary red wines, the addition of alcohol is decided by the relation of the alcohol to the extract (sugar deducted) exceeding sensibly the relation of 4 to 4.5. In Germany the relation of alcohol to glycerine is relied upon, the maximum proportion allowed being 100 parts by weight of alcohol to 14 of glycerine and the minimum 100 to 7. Wines going above the maximum are condemned as having suffered an addition of glycerine, those going below the minimum as being fortified with alcohol. With "sweet wines" these figures do not apply, as they are based on natural wines made in Germany. Moreover, no definite stand is taken upon the question of the fortification of the sweet wines from other countries sold in Germany. The Bavarian chemists require the content of alcohol and sugar in sweet wines used for medicinal purposes to be shown on the label, a very excellent provision, for no two samples of sweet wine can be depended upon to be of similar composition, and the physician is altogether in doubt as to what sort of a compound he is administering to his patient under the name of "port" or "sherry." Any wine with a higher percentage of alcohol than 15 per cent. by volume (12 per cent. by weight) can be safely declared to be fortified, for it has been shown that fermentation is arrested when the alcoholic content reaches about that point.

The *preservative agents* added to wine are entirely similar to those used in malt liquors. The subject of the use of salicylic acid has been so thoroughly investigated in the portion of the bulletin devoted to beer that a further treatment of the subject is unnecessary. The methods for detection and estimation already given for beer are applied equally as well to wine. The same may be said of sulphites and borax.

Mineral additions to wine are generally introduced accidentally, the strong acidity of the liquid making it very liable to contamination from metallic vessels, pipes, &c. Lead oxide was sometimes added to wine to counteract excessive acidity in former days, and Hassall gives cases of deaths traced to the use of such wines. Such additions belong probably to the adulterations of the past, although the possibility of

such a contamination should never be forgotten, especially if any of the symptoms of lead poisoning have been produced by a suspected sample. The search for mineral constituents in wine presents no difficulties and need not be further dwelt upon.

Gummy substances are sometimes added to watered wines to make up for their deficiency in extract. Gum arabic, or commercial dextrin, have been used for this purpose. The addition may be detected by the following method, taken from the German Commission: "10cc. of 95 per cent. alcohol are added to 4cc. of wine and the whole well shaken; if gums are present the liquid becomes milky and does not clear up completely even after standing several hours. The precipitate formed adheres in part to the sides of the glass, and forms lumps. In genuine wines flakes form after a short time, which subside and remain rather loose. For a more exact test the wine should be evaporated to a sirup, extracted with alcohol, and the insoluble part dissolved in water. This solution is treated with 1cc. hydrochloric acid, heated under pressure for two hours and the reducing power ascertained with Fehling's solution. With genuine wines no considerable reduction is obtained in this way." I have made no trial of this method.

The adulteration of wines by substituting for it, wholly or in part, the fermented juices of other fruits, such as cider, is a matter difficult of detection. The presence in such wines of malic acid and the absence of tartaric was formerly considered a sufficient proof of the addition, but it is found that in bad years malic acid often predominates in grape juice, and on the other hand various causes may greatly reduce the content of bitartrate of potash, or even cause it to entirely disappear. The proof of such addition by chemical means rests chiefly upon conclusions drawn from the general composition of the sample analyzed. Often the taste or odor of the residue of evaporation of the sample itself or of the distillate will give some clew to such addition. Often the recognition of free tartaric acid in such wines will condemn them as artificial, for natural wines contain a very small amount, if any, of the free acid; according to the German Commission never more than one-sixth of the total free fixed acids. Tartaric acid is often added also to wines which have been deprived of part of their normal acidity by the addition of water or sugar solutions. The qualitative determination is as follows:

To 20 or 30cc. of wine is added precipitated and finely powdered bitartrate of potash, the whole well shaken and filtered after standing an hour. To the clear solution is added 2 or 3 drops of a 20 per cent. solution of acetate of potash and the whole allowed to stand twelve hours. The shaking and standing of the solution must take place at as nearly as possible the same temperature. If at the end of this time any considerable precipitate has separated out, the quantitative estimation should be undertaken.

Foreign coloring matters are frequently added to red wines, either to brighten and improve the color obtained from the grapes, or, more frequently, to cover up the effects of previous dilution. These colors may

be of vegetable origin, obtained from the various vegetable dyes, or by mixing the juice of other highly colored berries or fruits with the wine; or they may be some of the numerous varieties of aniline dyes obtained from coal-tar. A few examples of the vegetable dyes said to be used may be mentioned as follows: Logwood, cochineal, elderberries, whortleberries, red cabbage, beet-root, mallow, indigo, &c.

Very elaborate and extensive schemes for the detection of these coloring matters have been devised, and chemical literature is full of articles written upon the subject, yet the positive identification of any of the vegetable coloring matters used is only very exceptionally carried out. Most of these schemes are based upon the difference in the color of the precipitates given with various reagents, and the coloring matters of the grape resemble so very closely in their behavior others of vegetable origin, and the variations in the amount of tannin present has so great an influence upon the character of the precipitate, that definite conclusions are well-nigh impossible.

The Berlin commission rejects all methods for the detection of vegetable coloring matters as not being capable of yielding positive proof, and gives only methods for the detection of coal-tar colors. The Paris Laboratory, on the other hand, gives a very elaborate scheme for the detection of both vegetable and aniline colors, designed to cover all substances likely to be used for such purposes. This scheme is based chiefly upon Gautier's and the French authorities claim that with it a chemist who is expert by long experience can detect the coloration of a wine by either vegetable or mineral foreign matters, though he may not perhaps be able in all cases to identify the particular coloring matter used. These schemes can only be referred to here, as I consider that their value is not sufficient to justify their reproduction.

The detection of aniline coloring matters can be made with tolerable certainty. The following method is essentially that given by the German commission, and originally devised, I believe, by König. Two samples of 100cc. each of wine are taken, and shaken up with about 30cc. of ether, after one has been rendered alkaline by the addition of 5cc. of ammonia. After separation has taken place, about 20cc. of the clear ethereal solution from each test are poured off (not filtered) and evaporated spontaneously in porcelain dishes in which are placed threads of pure white wool, about 5 cm. in length. With wines which are free from aniline colors, the wool, with the residue of the ammoniacal solution, remains of a perfectly white color, and the thread in the solution which was not treated with ammonia will be of a brownish color. The presence of fuchsine is readily detected, however; for out of the perfectly colorless ammoniacal ether solution a bright red color will appear as it evaporates, and becomes fixed upon the woolen thread. Those varieties of aniline dyes, which are more readily taken up by ether from acid solutions than from alkaline will be detected by the red coloring of the wool in the ether from the sample which received no addition of ammonia.

The coloring matter may also be extracted by means of amyl alcohol, which color will be discharged from the solvent by ammonia if the aniline dye used be of an acid nature, in which case the amyl alcohol will dissolve little coloring matter from the wine in presence of ammonia.

The *diseases* of wine may be considered in the light of an adulteration, as it is a fraud to offer wines for sale as pure wines which have undergone a change which alters their composition and renders them unfit for use. The researches of Pasteur on fermentation have shown that nearly all of the diseases of wine are due to the development in them of microscopical vegetable growths, whose germs are carried in the air. Each disease has its own special organism peculiar to itself, which may be detected by the microscope. These different organisms produce the souring, molding, bittering, cloudiness, blackening, &c., of wine. The best wines are said to be the most subject to these alterations; every year large quantities of the finest wines of Burgundy are spoiled by the disease called bittering (*l'amer*).

In wines that have become entirely unfit for use through the development of one of these diseases the fact is rendered sufficiently evident by the senses, especially to an expert taster. To detect the first beginning of such alteration, however, is more readily done by means of the microscope in the hands of an expert.

EXAMINATION FOR ADULTERATION OF THE WINES ANALYZED BY THE DEPARTMENT.

In the absence of any well-defined national standard as to what shall constitute a pure wine in the United States, or definitions and limitations as to the nature of the liquids which can lawfully be sold as such, I have had recourse to the well-defined and carefully worded laws of Germany and France which deal with the adulteration of wines, some of which, together with the accepted methods for the detection of adulteration, as agreed upon by chemists of prominence in those countries, I have collected together and inserted at the close of the Bulletin, under the heading of Appendix B.

The only State law I have been able to find which deals specifically with wine is a recent enactment in New York, which is also given in full in Appendix C.

The nature and extent of the different kinds of adulteration as shown by the samples examined may conveniently be taken up in the same order as was pursued in treating of the methods for detecting them, and of these the first is the dilution or watering of wine.

THE DILUTION OR WATERING OF WINE.

It would seem natural that in American wines, which can be produced so cheaply and in such great abundance, this adulteration, which is such a favorite one with the manufacturers of the costly wines of Bordeaux, Burgundy, &c., would be very rare. The fraud is so simple, however, so easy of execution, and so difficult of detection, that it will

probably always be a favorite one with unscrupulous dealers. It must be remembered, also, that with many American producers, whatever article they produce, more attention is paid to its quantity than its quality. Wine-growers are not the only persons who practice this method, as it can be done also by merchants and retailers, although in the latter case it is much more easy of detection. That which might be called scientific dilution, by means of the processes already described (petiotization, &c.), is much more difficult of detection than the simple attenuation of the wine by the retailer. So little official supervision has been exercised over the wines sold in this country that certainly the fear of detection has not operated very largely as a preventive of this, or in fact any other adulteration.

In Dr. Baumert's work, which has already been alluded to,¹ and to which I shall have occasion to refer frequently as constituting, small as it is, the only published investigation of American wines for adulteration, none of the samples fell below the German standard in percentage of extract (1.5 grams per 100cc.). On the other hand, nearly all the white wines which I submitted to a complete analysis fell below this standard, and two of the red wines. A large number of the samples analyzed by Mr. Parsons also fell below it. That this limit is not placed at too high a figure, for California wines at least, seems evident from a study of the table I have prepared of Professor Hilgard's analyses of pure wines, from which it appears that only one series of analyses gave a minimum below it, while the averages are far above it. It might possibly be too low for Virginia wines, but the majority of those that fell below it were of California origin. The New York law specifies (§ 2) that "such pure wines shall contain at least 75 per centum of pure grape or other undried fruit juice." Just how a chemist, in the absence of legal definitions of what shall constitute a "pure grape or other undried fruit juice," is to decide upon the question of such adulteration by the above law is difficult to indicate.²

The samples which would be considered as watered according to the German standard are as follows: Serial Nos. 5084, 5099, 4997, 4998, 5081, 5083, 5089, 5097, and 5098.

PLASTERING.

American wines would seem to be quite free from this form of adulteration. Baumert found no undue excess of sulphates in the samples he examined, but refers to a sample analyzed by Stutzer, which contained in 100cc. .141 gram SO_3 . In my seventy samples I found none which exceeded the generally adopted standard of .092 gram SO_3 to 100cc., or 2 grams K_2SO_4 to the liter, and only three, Nos. 5100, 5107, and 5115, which contained SO_3 , corresponding to over 1 gram K_2SO_4 per liter.

¹ Page 339.

² It will be seen by the above that cider would be considered as "wine" under the construction of the law, as it is the fermented juice of "other undried fruit."

FORTIFICATION.

It is evident that the German standard of 100 parts of alcohol by weight to 7. of glycerine, which is relied upon as a means of detecting the addition of alcohol, cannot be applied to American wines. Only three of the samples would pass muster by it, and it seems hardly possible that the practice of adding alcohol could be so widespread as would be thus indicated.

Below is given the number of grams of glycerine for 100 grams of alcohol obtained in the samples (exclusive of the sweet wines):

4995.....	7.1	5104.....	3.5
4996.....	5.6	4997.....	3.7
5005.....	9.0	4998.....	4.7
5084.....	6.7	4999.....	7.3
5088.....	4.5	5000.....	6.3
5094.....	5.7	5081.....	3.7
5095.....	3.4	5083.....	4.3
5096.....	4.3	5089.....	6.5
5099.....	5.0	5097.....	5.6
5100.....	3.1	5098.....	3.4
5101.....	6.3		
5103.....	3.7	Average.....	5.1

Baumert obtained very similar results; out of thirteen analyses (including sweet wines) made or collected by him only four contained a larger proportion of glycerine to alcohol than 7 to 100. Unfortunately no determinations of the glycerine were made in the pure wines analyzed by Hilgard; so no light is thrown on this point by them. The only possible way of deciding it, together with other questions relating to the composition of American wines, would be by the analysis of a large number of wines known to be pure. In the absence of such evidence, it would be useless to attempt to pass judgment on the above samples as to whether they had been fortified with alcohol or not.

The New York law allows of an addition to wine of "pure distilled spirits to preserve it" not to exceed 8 per cent. of its volume, which, supposing the wine to contain originally 10 per cent. by volume, would give a wine containing at least 17 per cent. by volume, or about the highest amount of alcohol which could be formed in a fermented liquor.

The sweet wines are, of course, well known to be fortified; they will be treated of more fully further on.

PRESERVATIVES.

Especial attention has been given in the present investigation to the use of improper preserving agents in fermented drinks. It was thought that such agents were much used; so a considerable number of samples were purchased, and the examination for preservatives, as well as for other adulterations whose detection did not require a complete analysis of the wine, was extended to all. The results show the practice to be even more extensive than was supposed.

The following table shows in what samples salicylic acid and sulphites were detected. In the case of the sulphites, where a "trace" is

indicated, there was not sufficient to justify the assertion that a sulphite or sulphurous acid had been added directly to the wine; in such cases it probably came from insufficient cleansing of the casks. Where it is indicated as "present," however, there was sufficient indication of its having been added to the wine.

Examination of wines for preservatives.

Designation.	Made in—	Serial number.	Salicylic acid.	Sulphites.
Champagne.....	New York.....	4960	None.....	None.
Do.....	do.....	4961	do.....	Do.
Do.....	Ohio.....	4962	do.....	Do.
Do.....	do.....	4963	do.....	Present.
Burgundy.....	do.....	4964	do.....	None.
Virginia seedling.....	do.....	4965	do.....	Do.
Catawba.....	New York.....	4966	Present.....	Do.
Sweet Scuppernong.....	North Carolina.....	4967	None.....	Do.
Charbono.....	California.....	4968	do.....	Do.
Lenoire.....	do.....	4969	do.....	Do.
St.-Macaire.....	do.....	4970	do.....	Do.
Angelica.....	do.....	4994	do.....	Do.
Burgundy.....	do.....	4995	do.....	Trace.
Claret.....	do.....	4996	do.....	None.
Moselle.....	do.....	4997	do.....	Present.
Riesling, Gray.....	do.....	4998	Present.....	None.
Riesling, Johannisberg.....	do.....	4999	do.....	Present.
Sauterne.....	do.....	5000	None.....	Do.
Sherry.....	do.....	5001	Present.....	None.
Sweet Burgundy.....	do.....	5002	None.....	Do.
Sweet Muscatel.....	do.....	5003	do.....	Do.
Tokay.....	do.....	5004	do.....	Do.
Zinfandel.....	do.....	5005	do.....	Do.
Catawba.....	do.....	5081	do.....	Trace.
California Hock.....	do.....	5082	do.....	Present.
California Riesling.....	do.....	5083	do.....	Trace.
Burgundy.....	do.....	5084	do.....	None.
Zinfandel.....	do.....	5085	do.....	Do.
St.-Julien Claret.....	do.....	5086	do.....	Trace.
Sweet Catawba.....	New York.....	5087	do.....	None.
St.-Julien Claret.....	do.....	5088	Present.....	Trace.
Riesling.....	do.....	5089	None.....	Present.
Sherry.....	California.....	5090	do.....	None.
Port.....	do.....	5091	do.....	Do.
Muscatel.....	do.....	5092	do.....	Do.
Angelica.....	do.....	5093	Present.....	Do.
Claret.....	Virginia.....	5094	None.....	Do.
Zinfandel.....	California.....	5095	do.....	Do.
Claret.....	Virginia.....	5096	do.....	Trace.
Riesling.....	California.....	5097	do.....	Present.
California Berger.....	do.....	5098	do.....	None.
Claret.....	Virginia.....	5099	do.....	Do.
Do.....	New Jersey.....	5100	Present.....	Do.
Do.....	Virginia.....	5101	None.....	Do.
Catawba.....	do.....	5102	Present.....	Do.
Claret.....	California.....	5103	None.....	Do.
Do.....	do.....	5104	do.....	Do.
Do.....	Virginia.....	5105	do.....	Do.
Sauterne.....	do.....	5106	do.....	Present.
Hock.....	do.....	5107	Present.....	Do.
California Beaune.....	do.....	5108	do.....	None.
Sweet Catawba.....	do.....	5109	do.....	Trace.
California Gutedel.....	do.....	5110	do.....	Present.
Claret.....	Virginia.....	5111	None.....	Do.
California Zinfandel.....	do.....	5112	do.....	Trace.
California Port.....	do.....	5113	do.....	None.
Sonoma Port.....	do.....	5114	Present.....	Do.
California Angelica.....	do.....	5115	do.....	Do.
Frontignan.....	California.....	5116	None.....	Do.
Old Pale Sherry.....	do.....	5117	do.....	Do.
California Zinfandel.....	do.....	5118	Present.....	Trace.
Gutedel Hock.....	California.....	5119	None.....	Do.
Berger Hock.....	do.....	5120	do.....	Present.
California Burgundy.....	do.....	5121	Present.....	Trace.
California Madeira.....	do.....	5123	do.....	None.
California Port.....	do.....	5124	None.....	Do.
California Tokay.....	do.....	5125	Present.....	Do.
California Frontignan.....	do.....	5126	None.....	Do.
California Angelica.....	do.....	5127	do.....	Do.
California Berger Hock.....	do.....	5128	do.....	Present.

From an examination of this table it will be seen that of the seventy samples examined, *eighteen*, or over one-fourth, had received an addition of salicylic acid, and *thirteen* had been preserved by the use of sulphurous acid, either as such, or in the shape of a sulphite. In two cases both agents had been used. One of the samples which contained salicylic acid and also one containing sulphites were among the samples exhibited at the meeting of the National Viticultural Convention last year in Washington.

The question of the propriety of the use of preservatives has been very fully discussed in previous pages, and will not be further enlarged upon here.

Baumert found no salicylic acid in the samples examined by him, and only traces of sulphurous acid.

The examination of the samples for boracic acid gave such peculiar results that I hesitate about pronouncing positively upon them until I shall have had opportunity to investigate the matter more closely. The test with turmeric paper gave slight traces present in all but *two* of the thirty-six samples which were submitted to a complete analysis. With only a very few, however, could any test be obtained with the alcohol flame. Baumert obtained the same test in every one of the samples he examined. He seeks to account for this by the fact that plants have been known to assimilate boracic acid from the soil, and knowing that in some parts of California the soil contains considerable quantities of borax, he offers it as an explanation that it was taken up by the vine from the soil. This explanation seems hardly tenable, and in view of the fact that some of the samples I examined came from various parts of the country other than California, must be thrown aside as insufficient. It is a singular fact that both of the samples in which I failed to get the test, Nos. 5087 and 5102 were Catawba wines.

I can offer no explanation of the matter except the suspicion, which I hope to be able to investigate, that the trouble lies with the test.

ARTIFICIAL WINES.

No test for free tartaric acid was obtained with any of the samples, which would seem to indicate that none of the wines were artificial wines, in the make-up of which free tartaric acid is very apt to figure.

COLORING MATTERS.

All of the samples of *red* wines, about forty, were submitted to a search for aniline coloring matters, which resulted in the demonstration that one sample out of the forty, No. 4996, was colored with an aniline dye-stuff, probably fuchsine.

Baumert found one of his eight samples to be colored artificially with an aniline dye.

No search was made for foreign *vegetable* coloring matters.

SWEET WINES.

It would seem advisable to call attention to the very variable character of these wines as shown by the analyses. Considering the extensive use that is made of such wines for medicinal purposes, it is greatly to be desired that some standard should be required for their composition, or that their relative content of alcohol and sugar, at least, should be stated on the label, as is required by the Bavarian authorities. Among Mr. Parsons' samples will be found a "Sweet Muscatel" which contains as high as 31 per cent of sugar, and a "California port" which contains nearly 21 per cent. of alcohol by weight. The analyses of the sweet wines made by myself furnish a still poorer showing, for the low figures obtained for glycerine show that very little pure grape juice enters into their composition. Take the different samples of Angelica and Muscatel wines, for instance; these varieties are almost peculiar to California; they are made from a very sweet grape, of strong flavor. Comparatively few analyses have been made of them, but Baumert had among his samples two Muscatel wines and one Angelica. These contained the following percentages of glycerine: Muscat (H), .883; muscat (W), 1.424; Angelica, .698.

Compare these numbers with the percentage of glycerine contained in the following: 5003, Muscatel, .102; 5092, Muscatel, .104; 4994, Angelica, .140; 5093, Angelica, .052.

These results are so disproportionately low as to give strong ground to the suspicion that but very little of the pure juice of these strong flavored grapes entered into the composition of the samples I examined, but that they were chiefly composed of alcohol, sugar, and water. It would be an easy matter to imitate the strongly marked flavor of the grapes by means of artificial essences.

California wine-growers claim that they have in their very pure grape brandy an excellent and unobjectionable source of alcohol for the fortification of sweet wines, but certainly the samples above partake more of the nature of a liqueur, than of a natural wine.

The following table gives a classification of the wines analyzed in the Paris Municipal Laboratory during the years 1881 and 1882, showing the proportion which was declared adulterated, and the relative amount of the different varieties of adulteration as shown by the samples analyzed. It must be remembered that these analyses were made on suspected samples, and do not by any means represent an average of the quality of the wines sold in Paris.

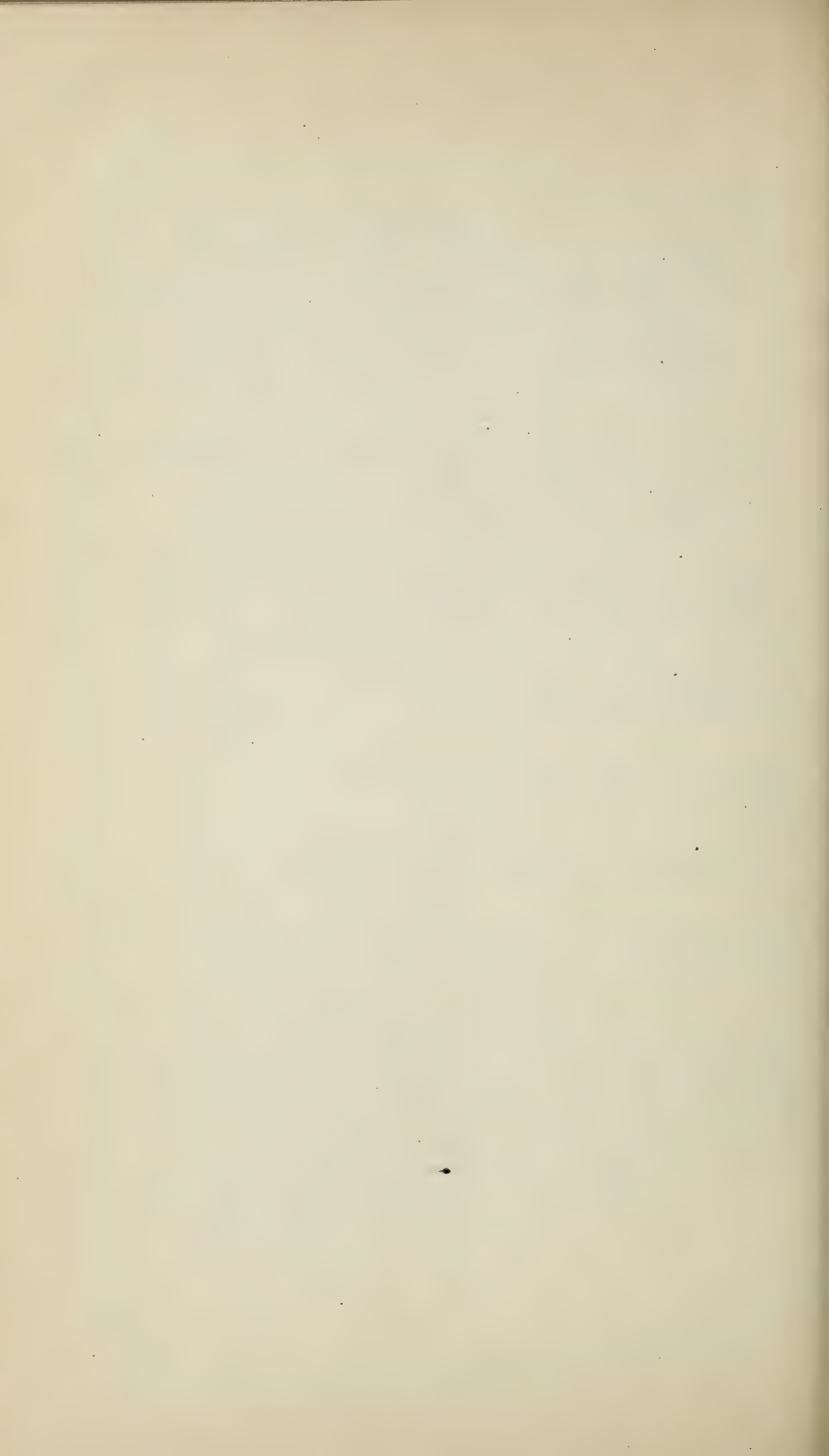
	1881.	1882.
Number of samples analyzed	3,361	5,150
Good	357	898
Passable	1,093	1,590
Diseases of wine (acid, bitter, moldy, &c.)per cent..	6.51	5.24
Fortified	9.55	7.32
Wines not plastered or plastered less than 1 gram.....do....	24.45	25.47
Wines plastered between 1 and 2 grams.....do....	52.53	41.49
Wines plastered above 2 grams.....do....	23.02	33.04
Watered	41.12	29.15
Sugared and petiotized.....do....	3.30	6.62
Artificially colored.....do....	15.65	7.66
Salicylated	4.73	5.00
Salted	0.18	0.08
Deplastered	0.11

A wine may be counted several times in this table; for instance, if it is at the same time watered, fortified, too much plastered, and artificially colored. The total of the percentages, therefore, adds up to over 100.

The samples analyzed by me may be tabulated as follows:

Total number analyzed.....	70
Plastered between 1 and 2 gramsper cent..	4.38
Watered (according to European standards).....do....	12.85
Artificially colored.....do....	1.43
Salicylated	25.71
Sulphured.....do....	18.57

The percentages are of the total number analyzed; not of the number adulterated, as in the French wines.



PART III.

CIDERS.



CIDER.

Cider is the fermented juice of the apple. It is an article of very general use, especially in those parts of the country where fruit-growing is carried on. Statistics of the amount produced or consumed are rather difficult to obtain, and I am unable to present any definite statement on the subject. It is quite a favorite article of home production, nearly every farmer in regions where apples are grown making his barrel of cider for use through the winter; but a large amount also finds its way into the city markets, finding ready purchasers among people who still retain their taste for the drink, acquired during a childhood on the "old farm." A considerable quantity is also consumed in the shape of bottled cider, "champagne cider," "sparkling cider," and similar substitutes for, or imitations of, champagne wine, large quantities of this clarified cider being produced in some parts of the country, notably New Jersey. Most of the cheaper kinds of champagne (American champagne) are made in this way.

In England and France considerable quantities of cider find their way into the markets, though it is there, as here, largely an article of home consumption. Certain parts of those countries are famous for the quality of their ciders, notably Normandy, in France, and Herefordshire and Devonshire, in England. France produced, in 1883, 23,493,000 hectoliters (620,211,200 gallons) of cider, or over one-half of the quantity of wine produced, and three times as much as the total quantity of malt liquors.

MANUFACTURE OF CIDER.

In the numerous sections of the United States where apples are grown in large quantities the manufacture of cider furnishes a most important means for the utilization of such fruit as is unfit for marketing, either from being too small or sour, or too thoroughly ripened, or bruised from handling. The conversion of these into cider, and perhaps of the cider into vinegar, is a very important branch of apple growing, and the cider press is an indispensable adjunct to a large orchard. Within the last ten years the manufacture of cider has been greatly aided by improvements, both in the machinery for crushing the fruit and in the presses for extracting the juice, but it is doubtful if the methods of treatment of the juice after extraction have undergone a corresponding development. The methods of fermentation and preserving—operations that are so carefully performed in the manufacture of other fermented

liquors—are exceedingly crude, as I can testify from personal experience. The juice, whether containing a relatively large percentage of sugar or not, is drawn into barrels and left to itself, probably exposed to a hot sun and to all the changes of temperature incident to the autumn season; and when the season is over or the cider is in danger of freezing, it is transferred to the cellar in the same barrels in which it was originally run, without any attempt at cleansing it of sediment, or filtering or racking, and when any attempt at improving its keeping quality is made it is by adding some antiseptic instead of freeing it from the matters which conduce to improper fermentations, or so conducting the process as to produce a liquor which can properly be called the “wine of apples.” It seems remarkable that with these methods so palatable a drink is produced, a fact which only shows what might be done if a little care and scientific knowledge were applied to the treatment of the juice. There is a great difference between the practice here and in other countries in regard to the treatment of the juice. Here the greater part of the cider produced is treated as indicated above, and is sold to the consumer in the fall or winter of the same year it is produced, without any treatment whatever, except perhaps the addition of a dose of mustard seeds or sulphite of lime or salicylic acid, to arrest or retard the fermentation. This addition serves only to stop the fermentation for a while, probably through the winter, and in the spring whatever has not been consumed has to be thrown away or turned into vinegar. In England and France the juice is treated according to the sweetness of the apples from which it is made, very sweet juice requiring a low temperature for its fermentation in order that the operation shall not be too rapid. The juice is run into barrels or large vats, which are kept in a barn or cellar where the temperature is more or less constant, and the fermentation allowed to go on until a “chapeau” or head of scum forms on top, containing many of the impurities of the juice. The clear liquid is then “racked off” from between the impurities which have risen to the top and those that have fallen to the bottom. The casks into which it is received are scrupulously clean and are filled nearly full and transferred to a cooler cellar, where a second slow fermentation takes place. The racking-off process may be repeated if necessary, or the juice may be filtered from the first fermentation. Cider fermented and properly racked in this way will keep indefinitely at a low temperature, especially if bottled. For bottling, it generally undergoes the operation called “fining,” by the addition of isinglass, which removes most of the albuminous constituents which are so inimical to its proper preservation. Cider made in this way will be much richer in alcohol, and contain much less acetic acid than when its first fermentation is allowed to take place at a high temperature and in a rapid, tumultuous manner. It is a true apple wine and will keep indefinitely. The cider of Devonshire has been kept twenty or thirty years.

COMPOSITION OF CIDER.

The amount of chemical work done on cider is not nearly so great as has been done on wine. In fact the published analyses of cider are very few and are confined almost entirely to other countries. I have not been able to find a single published analysis of American cider.

The following analysis of Alsatian cider was made by Boussingault, and dates back to 1863 :

	Grams per liter.
Alcohol	69.95
Sugar	15.40
Glycerine and succinic acid	2.58
Carbonic acid	0.27
Malic acid	7.74
Acetic acid	Traces.
Gummy matters	1.41
Potash	1.55
Lime, chlorine, &c	0.20
Nitrogenous matter	0.12
Water	980.78

Rousseau has published the mean of twenty analyses of Brittany cider, but his results are so low that it is thought by French authorities that his samples had been watered :

Alcohol, per cent. by volume	2.5
Extract grams per liter	19.3
Sugar	2.5
Total ash	1.52
Ash soluble in water	1.17

The following are analyses of pure ciders from different parts of France, made in the Paris Laboratory; the figures are in grams per liter :

	Pure cider, 1877, Bois-Guillaume.	Pure cider, made in 1876, Yvetot.	Old cider.	Pure cider, 1878, Yvetot.	Pure cider, "gros cider," 1880, near Bayeux.	Cider No. 2.	Cider, 1st class.	Pure cider, "gros cider," 1880, Bagneux.
Alcohol, in weight per liter. . .	47.40	41.08	37.92	34.76	23.70	7.90	25.30	19.75
Extract dried at 100° C	57.60	30.90	20.90	61.30	53.20	69.70	81.20	63.80
Extract dried <i>in vacuo</i>	60.10	37.60	27.00	72.70	60.80	82.00	92.60	75.00
Total ash	3.50	2.50	2.50	3.00	2.60	2.54	2.30	2.80
<i>Analysis of the ash.</i>								
Phosphates insoluble in water	0.38	0.25	0.30	0.45	0.62	0.17	20.55
Carbonate of potash	2.23	1.40	2.00	1.80	1.51
Other alkaline salts	0.89	0.85	0.70	0.35	0.41
Reducing sugar	20.00	7.50	4.40	3.70	16.50	36.00	39.00	25.00
Acidity expressed as H ₂ SO ₄ ..	3.60	4.07	5.36	4.54	3.23	2.68	2.08
Acidity of the cider dried <i>in vacuo</i>	2.50	2.40	2.59	2.31	2.68	1.11	1.48

Of these samples the first four had undergone a good fermentation. They furnish the following average composition for the principal constituents :

Alcohol, per cent. by volume	5.2
Extract per liter, at 100° C	41.18
Sugar	8.90
Ash	2.87

The other four samples were partially unfermented, or sweet, ciders. Their average composition was as follows :

Alcohol, per cent. by volume	1.70
Extract per liter, at 100° C	66.98
Ash	2.56

From these means the Municipal Laboratory deduces the following as a type of composition for pure ciders :

Alcohol, per cent. by volume	5.66
Extract per liter, at 100° C	30.00
Ash	2.80

Recent analyses of pure ciders, from different parts of France, published by M. G. Lechartier,¹ have shown great variations from this type, and show the necessity for the examination of large numbers of samples from various parts of the country for the establishment of a proper standard of analysis.

ANALYSES OF SAMPLES BY THE DEPARTMENT OF AGRICULTURE.

Various conditions rendered it impossible to extend the investigation of ciders to a very large number of samples. It is hoped that an opportunity for a more extended study will present itself in the future.

The samples for the investigation were purchased in the city in the same way as the samples of wine and beer.

METHODS OF ANALYSIS.

The different determinations to be performed in an analysis of cider can be made by the same methods as are used in the analysis of beer and wine. These have been already sufficiently discussed, and a brief résumé showing the amount taken for determination, &c., in the samples analyzed will be all that is necessary.

The specific gravity was taken with the pycnometer.

The alcohol was determined in the distillate from 100cc., after previous neutralization of the free acid.

The total solids were determined by drying to a constant weight, 10 grams in the case of the sweet ciders, 25 to 50 grams in the more completely fermented samples.

¹Compt. Rend. 103, 1164.

The acidity was determined in 25 to 50cc. by titration with decinormal soda, and calculated as malic.

The sugar was determined with Fehling's solution, as indicated under beer.

The ash was determined by incinerating the residue from 50cc. in a muffle.

The albuminoids were determined by evaporating 25cc. in a *schälchen* and burning with soda lime.

The carbonic acid was determined in the bottled ciders by the method given under beer.

The polarization given is that of the the normal cider in degrees of the cane sugar scale. It was taken on a Laurent polariscope.

Analyses of ciders by United States Department of Agriculture.

Designation.	Serial No.	No. of analyses.	Specific gravity.	Alcohol by weight.	Alcohol by volume.	Total solids.	Free acids, as malic.	Sugar, as dextrose.	Ash.	Albuminoids.	Carbonic acid.	Polarization cane-sugar scale.
<i>Well fermented ciders.</i>												
Draft cider ("extra dry")	4830	1	1.0132	4.18	5.23	3.31	.602	(¹)	.396	.038	-19.5
Bottled cider, known to be pure	4832	2	1.0003	8.69	10.05	1.88	.456279	.063	trace	-7.0
Bottled cider	4833	3	1.0007	6.28	7.83	1.80	.376340	.044	-6.1
Bottled "extra dry russet" cider	4834	4	1.0264	4.48	5.61	5.52	.330393	.031	-35.2
"Champagne cider," bottled	4835	5	1.0223	4.08	5.10	5.02	.567310	.050	.161	-23.4
"Champagne cider," bottled	4836	6	1.0143	5.45	6.79	3.69	.361415	.038	.120	-20.4
"Sparkling cider," bottled	4927	7	1.0306	3.63	4.54	5.92	.113506	(²)	-33.8
Average	1.0154	5.17	6.45	3.88	.402377	.044
<i>"Sweet" or incompletely fermented ciders.</i>												
Draft cider	4829	1	1.0537	0.65	0.81	9.34	.565315	.069	-41.6
"Sweet" cider	4831	2	1.0516	0.61	0.77	9.59	.302270	.063	-34.2
"Sweet" cider (draft)	4837	3	1.0567	0.20	0.25	9.53	.375283	.075	-48.4
Do.	4838	4	1.0203	3.46	4.33	3.84	.302374	.044	-24.2
Do.	4839	5	1.0552	0.55	0.67	9.75	.409336	.031	-48.5
Do.	4841	6	1.0355	2.96	3.71	6.98	.478348	.069	-39.1
Average	1.0455	1.40	1.76	8.17	.405321	.059

¹A circumstance arising after the samples had been thrown away seemed to throw considerable doubt upon the determinations of sugar, which were made by an assistant, and the entire set had to be thrown out.

²Determinations of the carbonic acid in three different bottles gave the following results: .728, .654, .482.

ADULTERATION OF CIDER.

Cider is very little subject to adulteration according to most of the authorities on foods. Even Hassall, who generally enumerates under each article of food a list of every conceivable adulteration that has ever been found or supposed to have been used in such food, only speaks of the addition of water, of burnt sugar as a coloring matter, and of the use of antacids for the correction of the acidity of spoiled cider. On the other hand, in France, where, as we have seen, it is

very largely consumed, its adulteration is by no means uncommon, although principally confined to its watering, together with additions for the purpose of covering up such attenuation, such as foreign coloring matters. In the Paris Municipal Laboratory out of 63 samples examined in 1881, 39 were pronounced "bad," among which were 26 artificially colored; in 1882, 59 samples were examined, of which 30 were declared "bad," of which 7 samples were artificially colored; 2 samples contained salicylic acid. The following is considered there as a minimum limit for the composition of a pure cider, and any sample which falls below it in any constituent is considered as watered:

Alcohol, per cent. by volume	3.
Extract in grams, per liter.....	18.
Ash.....	1.7

This is for a completely fermented cider; in sweet ciders the content of sugar should exceed the limit sufficiently to make up for the deficiency of alcohol, to which it should be calculated.

EXAMINATION OF THE SAMPLES FOR ADULTERATION.

The investigation of the samples was undertaken with the full expectation of finding a considerable number preserved with antiseptics. This supposition failed to be confirmed, however, for no salicylic acid was found, and in but one case was any test obtained for sulphites. None of the samples fell below the standard proposed by the French chemists, given above, and no metallic or other adulteration was discovered.

The single exception, however, No. 4927, was an embodiment in itself of nearly all the adulterations which have been enumerated as possible in cider. It was handsomely put up in neatly capped bottles, and of a clear, bright color. Its tremendous "head" of gas when uncorked gave rise at once to the suspicion that it had received some addition to produce an artificial pressure of gas, for pure cider does not contain sufficient sugar to produce very much after-fermentation, any more than pure wine. The low content of free acid, together with the large amount of ash and very variable content of carbonic acid in different bottles, established the fact that bicarbonate of soda had been added, probably a varying quantity to each bottle, while the dose of sulphites added was so large that a bottle has stood open in the laboratory all through the summer without souring.

To describe in detail the methods for the detection of the adulterations of cider would be simply a repetition of what has been previously given in connection with either beer or wine; the search for preservatives is precisely the same, and the detection of the addition of antacids has been fully treated of under beer. No search was made for artificial coloring matters, as it seems very improbable that any such should be used in this country, and no evidence of any dilution was found.

APPENDIX A.

Since the portion of this bulletin relating to malt liquors was written, a bill has been introduced into the British Parliament dealing with the question of the use of substitutes for hops and malt in beer brewing; the text of this bill is as follows:¹

A BILL for better securing the purity of beer. (A. D. 1887.)

Whereas it is expedient, with a view to enable the public to distinguish between beer brewed from hops and malt from barley and beer composed of other ingredients, to amend the law relating to the sale of beer :

Be it therefore enacted by the Queen's Most Excellent Majesty, by and with the advice and consent of the Lords Spiritual and Temporal, and Commons, in this present Parliament assembled, and by the authority of the same, as follows :

(*Short title.*) 1. This act may be cited as the Pure Beer Act, 1887.

(*Declaration of ingredients on selling beer.*) 2. Every person who sells or exposes for sale, by wholesale or retail, any beer brewed from or containing any ingredients other than hops and malt from barley shall keep conspicuously posted at the bar or other place where such beer is sold or exposed for sale, a legible notice stating what other ingredients are contained in such beer. Any person who sells or exposes for sale any such beer as aforesaid without complying with the above enactment shall be liable to a fine not exceeding in the case of the first offense *five pounds*, and in the case of the second or any subsequent offense *twenty pounds*. Any fine incurred under this section may be recovered summarily by any informer, and one-half of the fine shall in every case be paid to the informer.

(*Definition of beer.*) 3. In this act the term "beer" includes beer (other than black or spruce beer), ale, and porter.

(*Commencement of act.*) 4. This act shall come into operation on the *first day of January, one thousand eight hundred and eighty-eight*.

A BILL for better securing the purity of beer. (A. D. 1887.)

Whereas it is expedient, with a view to the better protection of the public from adulteration of beer, to amend the law relating to the sale of beer :

Be it therefore enacted by the Queen's Most Excellent Majesty, by and with the advice and consent of the Lords Spiritual and Temporal, and Commons, in this present Parliament assembled, and by the authority of the same, as follows :

(*Short title.*) 1. This act may be cited as the Beer Adulteration Act, 1887.

(*Penalty on selling beer containing other ingredients than hops and malt without giving notice.*) 2. Every person who sells or exposes for sale by wholesale or retail any beer brewed from or containing any ingredients other than hops and malt from barley, shall keep conspicuously posted at the bar, or other place where such beer is sold or exposed for sale, a legible notice stating that other ingredients are contained in such beer.

¹ Analyst, 1887, 99.

Any person who sells or exposes for sale any such beer as aforesaid, without complying with the above enactment, shall be liable to a fine not exceeding in the case of the first offense *forty shillings*, and in the case of the second or any subsequent offense *ten pounds*.

Any fine incurred under this section may be recovered summarily by any informer, and one-half of the fine shall in every case be paid to the informer.

(*Definition of beer.*) 3. In this act the term "beer" includes beer (other than black or spruce beer), ale, and porter.

(*Extent of act.*) 4. This act shall not extend to Ireland.

(*Commencement of act.*) 5. This act shall come into operation on the first day of January, one thousand eight hundred and eighty-eight.

This bill naturally excited considerable interest among food analysts, to whom would fall the duty of determining the question that would arise, under its enforcement, as to whether beers had been made from hop or malt substitutes, and a circular was sent out by the president of the Society of Public Analysts to the members of the society drawing their attention to the bill and asking them to report to the secretary any information they might have on the points whether the substances used as substitutes for hops could be detected and identified with certainty by chemical analysis, and what opinion, if any, they might have as to the effect on health of habitual small doses of such hop substitutes.¹

This circular called forth a few responses, among them the following paper by Mr. Allen:²

AN IMPROVED METHOD OF DETECTING QUASSIA AND CERTAIN OTHER HOP SUBSTITUTES IN BEER.

[Read at the meeting in May, 18-7.]

Hitherto the detection of hop substitutes in beer has had for English analysts little more than an academic interest. There has been no definition of beer, nor standard of strength or quality, and hence the brewer has been free to employ any hop substitute that could fairly be regarded as non-injurious. Now, however, that there are two bills before Parliament, both of which aim at rendering the employment of hop substitutes illegal, unless duly acknowledged, the question has acquired considerable practical importance. If the bills in question ever become law, it will devolve on the public analyst to certify to the presence or absence of hop substitutes, but, as was recently pointed out by the chancellor of the exchequer to a deputation which waited on him, it would be of no use to pass an act forbidding the unacknowledged use of hop substitutes unless it was possible to detect infringement of the law. Hence it has become important for public analysts to see how far the more important hop substitutes can be definitely detected in beer, or at any rate distinguished from hops.

The problem in question is by no means a simple one. Beer itself is a highly complex and variable product, and some of the normal constituents add to the difficulty of detecting hop substitutes. The problem is further complicated by the possible presence of several substitutes simultaneously, together with actual hops. Then the bitter principles to which the hop substitutes owe their employment have in some cases been very imperfectly studied, and belong to a class of bodies by no means distinguished for strong chemical affinities or for characteristic reactions. In fact, the

¹Analyst 1887, p. 79.

²*Ibid.*, p. 107.

most general and striking property of the majority of hop substitutes is the intensity of their bitter taste, a character which materially increases the difficulty of detecting them, owing to the very moderate amount employed to give the beer the desired flavor. There is one other complication of practical importance, and that is the very considerable quantity of beer usually recommended to be used for the analysis. Thus, if two liters be used for the main examination, as is recommended by Dragendorff, at least twice that quantity should be submitted to the analyst, and hence twelve liters would have to be purchased. The necessity of purchasing so large a quantity as two and a half gallons of each beer would almost certainly render the act abortive.

In looking into the question, therefore, I have aimed at reducing the amount of beer employed as low as possible.

In the last number of the Analyst I gave a list of references to articles in English periodicals, &c., connected with detection of hop substitutes in beer. These I have carefully studied, and they have formed the foundation of the following statements and proposals:

Dragendorff, who has published a very elaborate method for the recognition of a large number of possible hop substitutes (Jour. Chem. Soc., XXVII, 818; XLII, 103), operates on two liters of beer, precipitates the concentrated liquid with basic acetate of lead, further concentrates the filtrate and adds a large volume (1,000 to 1,200cc.) of absolute alcohol. The spirit is subsequently driven off, and the various principles extracted by a systematic employment of immiscible solvents.

Wittstein (Jour. Chem. Soc., XXIX, 767) operates in a similar manner, but works on one liter, and omits the treatment with acetate of lead, as indeed did Dragendorff in his older method. In the method described by me in my Commercial Organic Analysis (Vol. I, page 97), based on a process of Ender's, one liter of beer is employed, the concentrated liquid is treated with alcohol, the filtrate precipitated with ether, and the filtered liquid evaporated, the residue redissolved in alcohol, treated with water, and the solution precipitated with acetate of lead.

Several of the writers on the subject state that on precipitating a beer with basic acetate of lead the hop-bitter is wholly precipitated, and hence, if the concentrated filtrate still have a bitter taste, the presence of some hop substitute is certain.¹

This difference seemed to me so important that I have very carefully investigated it, and find it perfectly in accordance with the fact. I prefer, however, to employ *neutral* acetate of lead instead of the basic or ammoniacal acetate, as the latter reagents are liable to precipitate certain bitter principles not removed by the first. On the other hand, the hop-bitter is very perfectly precipitated by neutral lead acetate, and this statement is equally true of an infusion of hops purposely prepared and of a beer in which hops is the sole bitter used; but I have some reason to think that basic acetate of lead is liable to effect a less perfect separation of the hop-bitter.

In attempting to improve these processes I have endeavored to dispense with the use of alcohol, and yet separate the bitter principles from the sugar and other bodies which disguise the bitter taste and interfere with application of chemical tests, and find that a very satisfactory product for further treatment can be obtained by the following simple method:

One liter of the beer is evaporated to about 300cc. and is then precipitated, while hot, with a solution of neutral acetate of lead. The precipitate is filtered off, the filtrate allowed to become cool, and any further precipitate is also removed. The excess of lead is then removed from the filtrate by sulphuretted hydrogen, and the liquid fil-

¹This distinction between the bitter principles of hops and hop substitutes is referred to in the work entitled *Chemistry Applied to the Arts and Manufactures*, edited by Chas. Vincent, and often described as the New Edition of Muspratt's Chemistry. The process is also described in Wynter Blyth's work on the Analysis of Food, and forms an essential part of Ender's method of detecting bitter substances, so that there is a very general consensus of opinion as to the value of the test.

tered and further concentrated to about 150cc. I prefer sulphuretted hydrogen to sulphuric acid or a sulphate, as the lead sulphide seems to carry down a notable quantity of coloring matter.

The treatment with lead acetate removes all tannin, phosphates, &c., and *the hop-resin and lupulin are also completely precipitated, while all or nearly all hop substitutes remain in solution.*

The next object is to separate the sugar, dextrin, and mineral constituents of the beer as perfectly as possible from the active principles of the various hop substitutes. Instead of precipitating the carbo-hydrates, &c., by excess of strong alcohol, I prefer to remove the bitters from the aqueous liquid itself by agitation with suitable immiscible solvents.

The object being to extract as many active principles as possible in the simplest possible way, leaving their mutual separation and recognition for further consideration, I employ chloroform as having the most general solvent action. It separates with tolerable ease from the aqueous liquid, and should be employed as long as it leaves a notably bitter residue on evaporation. In many of my experiments I relied on the traces of lactic and other acids naturally present in beer to produce the requisite degree of acidity, but I am now of opinion that the addition of a little dilute sulphuric acid is advantageous, if not actually necessary, in some cases. The extraction with chloroform being complete, ether should next be used, the treatment being repeated as long as any notably bitter principle is extracted. Finally, the aqueous liquid is rendered alkaline with ammonia, and agitated with chloroform or ether-chloroform, to extract any alkaloids.

The following arrangement shows the behavior of the more important bitter principles when the aqueous liquid is agitated in succession with chloroform, ether, and ammonia, and ether-chloroform. I have personally verified the behavior of the substances to the names of which asterisks are attached.

(1) Extracted by chloroform from acid solutions:

Absinthin (wormwood).

*Anthemini (chamomiles).

Colchicine (colchicum), imperfectly.

*Colocythin (colocyth, or bitter apple), imperfectly.

*Calumbin, and probably some berberine (calumba), bright yellow and highly fluorescent.

*Gentipierin (gentian), very imperfectly.

*Picric acid (artificial), yellow, imperfectly.

Picrotoxin (*cocculus indicus*), with difficulty.

*Quassini (quassia wood).

(2) Subsequently extracted by ether from acid solutions:

*Chiratin (chiretta).

*Colocythin (colocyth, or bitter apple).

*Gentipierin (gentian).

Picric acid, yellow.

Picrotoxin (*cocculus indicus*).

(3) Subsequently extracted by ether-chloroform from alkaline solutions:

*Berberine (calumba root).

Colchicine (colchicum).

By evaporating off the solvent, warming the residue with a little alcohol, and then adding water, solutions are obtained which will be bitter if any of the above substances be present. A very small quantity of the substance is required for this test; indeed, the use of too large an amount must be carefully avoided or the sense of taste will be found to be wholly paralyzed for the remainder of the day at any rate.

It will be seen that chloroform or ether extracts from acidulated aqueous liquids almost the whole of the above bitters. The subsequent treatment with ether-chloroform in alkaline solution is usually unnecessary, as the principles of calumba and

colchicum are in part extracted by acid chloroform. Seeing that the bitter principles of hops are entirely precipitated by neutral acetate of lead, *the presence of some hop substitute is absolutely certain if the chloroform or ether residue has a marked bitter taste.* This can be ascertained in the course of a few hours by the simple method above indicated, and half a pint of the beer is amply sufficient for the purpose.

The presence of a hop substitute being proved by the marked bitter taste of the chloroform or ether extract it will of course be very desirable to ascertain its nature, and in some important cases this can fortunately be effected very satisfactorily. In others we may expect future investigation to afford the necessary assistance.

I have made special endeavors to ascertain the possibility of definitely recognizing quassia, which is one of the most important of the hop substitutes actually employed.

For this purpose I prepared quassiin in a moderately pure state by exhausting quassia wood with hot water and treating the decoction with acetate of lead and chloroform, in the manner recommended for beer. The quassiin was obtained with some difficulty in a distinctly crystalline state and otherwise it presented a close general resemblance to the description of it given by other observers. The following characters and tests were specially verified :

Quassiin is intensely and persistently bitter, sparingly soluble in cold water, more readily in hot, and easily soluble in alcohol. Its best solvent is chloroform, which extracts it readily from acidulated solutions.

An aqueous solution of quassiin does not reduce Fehling's solution, or ammonio-nitrate of silver. The solid substance gives no coloration (or merely yellow) when treated with strong sulphuric acid or with nitric acid of 1.25 specific gravity, nor is any color produced on warming. These four negative reactions are important, for *pirotoxin* reduces Fehling's solution and gives an orange-red color with sulphuric acid; *gentipicrin* and *menyanthin* reduce ammonio-nitrate of silver, and the former gives a red color and the latter a yellowish brown, changing to violet red when warmed with sulphuric acid, and other bitters mostly give more or less characteristic reactions.

A solution of quassiin gives a white precipitate with tannin. The reaction is used by Christensen, Oliveri, and others to isolate quassiin from its solutions, and by Enders to separate it from *pirotoxin*. In my hands the reaction has not proved satisfactory. The liquid is very difficult to filter and the filtrate still retains an intensely bitter taste, showing that the precipitation is very incomplete. As an analytical method the reaction is useless, but it is of some value as a qualitative test.¹ The test must be made in a cold solution.

Quassiin gives a brown coloration with ferric chloride. The reaction is best observed by moistening a quassiin residue in porcelain with a few drops of a weak alcoholic solution of ferric chloride, and applying a gentle heat. A fine mahogany-brown coloration is produced.

The most delicate and characteristic test for quassiin is based on an observation of Christensen. On treating quassiin with bromine a derivative is obtained, which is stated to be more bitter than the original substance. On adding caustic soda the bitter taste is said to be destroyed, but a product of a fine yellow color is obtained. I am unable to confirm the destruction of the bitter taste, at least entirely, but the coloration is marked and characteristic.

The following is the best way of applying the test: The substance to be tested for quassiin is dissolved in a little chloroform, or if a liquid is agitated with chloroform, and the aqueous layer separated. The chloroformic solution is then treated with bromine water until the yellow color remains after agitation, showing that the bromine has been used in slight excess. The aqueous liquid is then removed (or if small in volume may be neglected), and the chloroform agitated with ammonia. This produces immediate destruction of the color due to the bromine, and if quassiin be ab-

¹ Possibly more complete precipitation of quassiin by tannin could be effected in an alcoholic solution.

sent both the chloroform and ammoniacal liquid will be colorless. In presence of quassia the ammonia will be colored a bright yellow.

The chloroform residues from camomiles, calumba, colocynth, cocculus, and chiretta do not give any similar reactions with bromine and ammonia. The ether residue from chiretta gives a straw-yellow coloration, gradually changing to a dull purplish brown, but the fact that no such reaction yielded by the chloroform solution of the drug renders confusion with quassia impossible. Picric acid yields a solution in chloroform which is but slightly colored compared with the deep-yellow liquid produced on subsequent agitation with ammonia; but if its presence be suspected it can be readily and completely removed by agitating the chloroformic solution with soda or ammonia, and separating the alkaline liquid before employing bromine.

With a view of ascertaining how far the foregoing reactions of quassia were likely to be of service in practice I added to one liter of a mild beer, which had been previously proved to yield no bitter principle to chloroform after treatment with acetate of lead, sufficient infusion of quassia to make a perceptible difference in the flavor. The liquid was concentrated, precipitated with neutral lead acetate, the filtrate treated with sulphuretted hydrogen, and the refiltered liquid further concentrated and agitated with chloroform. On evaporating the chloroform a residue was obtained which had an intensely bitter taste, and yielded a solution which gave a white precipitate with tannin, but did not reduce ammonio-nitrate of silver. The residue gave no color on warming with concentrated sulphuric acid, but gave a well-developed mahogany-brown color with ferrie chloride. By the bromine and ammonia test it gave a strong yellow coloration.

The amount of residue obtained would have sufficed to obtain all these reactions several times, so that it may be considered established that *quassia can be detected with certainty and facility in a moderate quantity of beer containing it.*

The employment of *chiretta* as a hop substitute has been repeatedly recorded by previous observers, but no tests are given for it by Dragendorff or others who have worked on the subject. I found it in quantity in two hop substitutes I recently examined, and suspect its presence in a third. The active principle (*chiratin*, $C_{25}H_{48}O_{15}$) is intensely bitter, sparingly soluble in cold water, rather more so in hot, and is readily dissolved by alcohol and ether, the latter solvent readily removing it from its aqueous solution. On the other hand, chloroform removes but little bitter principle from an aqueous infusion of *chiretta*. *Chiratin* is a neutral substance, decomposed by dilute acids into *ophelic acid* and *chiratogenin*. It does not reduce Fehling's solution, gives a copious precipitate with tannin, and is not precipitated by neutral lead acetate. The reaction of the other residue from infusion of *chiretta* with bromine and ammonia has already been described.

It is evident that our knowledge of the chemistry of the vegetable bitters available as hop substitutes is very incomplete, and it is only by its further study we can hope to fully solve the problem of their detection in beer. But I believe we can already distinguish with certainty and facility between "hops" and "not hops," and that ought to suffice in many cases. When we examine butter we are content to define the admixture as "foreign fat," and we make no attempt to specify the exact nature or origin of the foreign fat employed. I submit that we are fully able to take a similar position with respect to hops and hop substitutes.

The following are some of the remarks made in the discussion of this paper:²

Dr. Adams said that he worked on the question of hop substitutes some ten or twelve years ago, and he found no difficulty in distinguishing between the bitter of hop and the substitutes used for it. The method he found most useful was the precipitation by subacetate of lead, and there was no difficulty at all with the ordinary bitters such as quassia, calumba, gentian, *chiretta*, and wormwood, all remaining in

²*Ibid.*, p. 114.

solution, whilst the bitter of hop, and also the bitter of the camomile, which behaves like the hop, goes down and leaves the solution bitterless. He had specimens of all the hop substitutes in use in England at that time, and without a single exception the solution remained bitter after treatment with the subacetate of lead.

After having separated the filtrate and evaporated it down, there was no difficulty in detecting the bitter; but he did not think there was any possibility of distinguishing between the individual bitters, considering the minute quantity present, and one could only positively say there was another bitter present besides that of the hop.

Dr. Muter said that, with regard to the question of the detection of bitters in beer, he would say at once that he did not quite share the president's views as to there being no difficulty in detecting and identifying them. It was a subject that he had worked at in former years in connection with his book on *materia medica*. He had made numerous experiments himself, and he had repeated many of the published experiments and he could prognosticate that, as the president went along, he would meet with several published reactions which were partially incorrect and perfectly misleading. Although a few bitters would be found to be comparatively easy of detection—quassia, for instance, was a bitter which spoke very much for itself—many of them were, to a great extent, involved in difficulty and obscurity at the present time. There was no branch of chemistry that so much undesirable matter had been published about as that relating to *materia medica*, and many of the older researches were incorrect. No doubt one cause of this was that nowadays they had much better analytical appliances than the men who made these experiments. He did not for a moment mean to say that they were now cleverer than past observers, but, for instance, they could not wash lead and other similar intractable precipitates in former days as one could now do by the aid of the filter pump, and the color reactions for more than one proximate principle, which were given in books, were really not due to the principle at all, but to the traces of reagents and other matters that remained with them owing to the imperfect washing, which was almost certain to occur before the days of filter pumps. He was afraid that until the president and others who might take the subject up had had time to work it out by the aid of modern appliances it would be going too far to say they could swear positively that a sample contained no hop substitute.

Mr. Allen said he thought they could tell whether it contained hop or a substitute for hops.

Dr. Muter then said in that case how would they get on in cross-examination, seeing that they could not name the substitute? He did not remember how he first became acquainted with the lead process, but he believed that he could put his hands on it now. It must be quite thirteen or fourteen years ago since it was first published. Immediately it was brought out he had made experiments upon it, working on large quantities. Since that there had really been no other process that he knew of. He himself had used a process very similar to that mentioned, viz, precipitation with subacetate of lead, siphoning off the clear liquor after settlement, removing the excess of lead from this liquid, concentrating and tasting, and then extracting with immiscible solvents.

Now came a difficulty which shook his faith in his powers as an analyst as regards hop substitutes. He had always believed in the process—from practicing upon beer with various added bitters—until some time ago he got a beer which he was privately assured by the maker to have no bitter other than hops. This sample he put through the process and he got a bitter out of that beer with chloroform after lead. He worked on a fairly large quantity, but the process here showed bitters other than hop, although he was assured that the sample represented as pure a beer as could possibly be obtained. Another difficulty was the quantity they might have to work on. Supposing an inspector brought them one-half or one-third of a pint, where was the process they could use? He had put bitters in the beer and worked on such quantities and failed to find them. In a case he knew of some time ago there was some

difficulty about some strychnine that was put in beer; he was aware of the very small quantity that had been put in, because the chemist who had been stupid enough to lend himself to such a transaction had informed him of it. He made up some beer and divided it into two portions, tried for extraneous bitters in one portion by the regular beer way and there was not a sign of it; he then tried the other portion with a special toxicological process for strychnine and found it. He had, even then, to use eight ounces of the beer for this purpose, to get a really satisfactory ordinary reaction. In the present state of chemical knowledge it would not be, in his opinion safe to say they could detect any amount of added bitters to beer, however small, and go to the length of naming those bitters on the quantity they would have usually brought to them by an inspector. With a gallon of beer and an unlimited fee covering many days' work they might, however, be able to do something satisfactory towards it.

Mr. Allen, in reply: If he had an insufficient quantity of any sample, he certified that the quantity was insufficient for him to form an opinion.

He understood Mr. Norton to consider it of great importance that they should be able to distinguish between "hops" on the one side and "not hops" on the other; this, he believed, could be done with certainty, ease, and on a very moderate quantity of beer. As to the identification of the various hop substitutes he did not profess to be able to distinguish all, but he thought he could already positively recognize calumba, quassia, colocynth, and some other bitters, including picric acid and picrotoxin, and if the matter became important he believed in a year or two public analysts would have devised methods for the detection of the other bitters, just as they had conquered other analytical difficulties when the occasion arose.

From the above it would appear that the lead method, which I employed, is considered by the English analysts as capable of deciding whether substitutes for hops have been used.

No action seems to have been taken as yet on the question of malt substitutes.

APPENDIX B.

As frequent references have been made in the body of the Bulletin to methods of analyses and manner of judging adapted by European chemist for beer and wine, I have thought it proper to give some of these methods complete. They represent the conclusions of chemists who have devoted their lives to the study of this branch of work, and may very properly be taken as a guide by those of us who have occasion to follow the same line of work in this country, where beer and wine analysis has as yet had little application.

The translation has been made as literal as possible.

The following are the methods adopted by the Berlin Commission:¹

A commission of experts, appointed in the year 1884 by the chancellor of the Empire, to which was intrusted the establishment of uniform methods for the chemical investigation of wine, adopted the following resolutions, which were made public by the Prussian minister for commerce and trade by a decree of the 12th August, 1884, which provides that they shall be rigidly adhered to in public institutions for the examination of food-stuffs, and are recommended to the representatives of like private concerns:

RESOLUTIONS OF THE COMMISSION FOR ESTABLISHING UNIFORM METHODS FOR THE ANALYSIS OF WINES.

Since, in consequence of improper manner of taking, keeping, and sending in of samples of wine for investigation by the authorities, a decomposition or change in the latter often occurs, the commission considers it advisable to give the following instructions:

INSTRUCTIONS FOR SAMPLING, PRESERVING, AND SENDING IN OF SAMPLES OF WINE FOR EXAMINATION BY THE AUTHORITIES.

(1) Of each sample, at least one bottle ($\frac{3}{4}$ liter), as well filled as possible, must be taken.

(2) The bottles and corks used must be perfectly clean; the best are new bottles and corks. Pitchers or opaque bottles in which the presence of impurities cannot be seen are not to be used.

(3) Each bottle shall be provided with a label, gummed (not tied) on, upon which shall be given the index number of the sample corresponding to a description of it.

(4) The samples are to be sent to the chemical laboratory as soon as possible to avoid any chance of alteration which, under some circumstances, can take place in a very short time. If they are, for some special reason, retained in any other place for any length of time, the bottles are to be placed in a cellar and kept lying on their sides.

¹ Das Gesetz betreffend den Verkehr mit Nahrungsmitteln, u. s. w., p. 184.

(5) If in samples of wine taken from any business concern adulteration is shown, a bottle of the water is to be taken which was presumably used in the adulteration.

(6) It is advisable, in many cases necessary, that, together with the wine, a copy of these resolutions be sent to the chemist.

A.—Analytical methods.

Specific gravity.—In this determination use is to be made of a picnometer, or a Westphal balance controlled by a picnometer. Temperature 15°C .

Alcohol.—The alcohol is estimated in 50–100cc. of the wine by the distillation method. The amount of alcohol is to be given in the following way: In 100cc. wine at 15°C . are contained n grams alcohol. For the calculation the tables of Baumbauer or Hebbner are used.

(The amounts of all the other constituents are also to be given in this way; in 100cc. wine at 15°C . are contained n grams.)

Extract.—For this estimation 50cc. of wine, measured out at 15°C ., are evaporated on the water bath in a platinum dish (85mm. in diameter, 20mm. in height, and 75cc. capacity, weight about 20 grams), and the residue heated for two and one-half hours in a water jacket. Of wines rich in sugar (that is, wines containing over 0.5 grams of sugar in 100cc.) a smaller quantity, with corresponding dilution, is taken so that 1 or at the most 1.5 grams extract are weighed.

Glycerine.—One hundred cubic centimeters of wine (for sweet wines see below) are evaporated in a roomy, not too shallow, porcelain dish to about 10cc., a little sand added, and milk of lime to a strong alkaline reaction, and the whole brought nearly to dryness. The residue is extracted with 50cc. of 96 per cent. alcohol on the water bath, with frequent stirring. The solution is poured off through a filter, and the residue exhausted by treatment with small quantities of alcohol. For this 50 to 100cc. are generally sufficient, so that the entire filtrate measures 100–200cc. The alcoholic solution is evaporated on the water bath to a sirupy consistence. (The principal part of the alcohol may be distilled off if desired.) The residue is taken up by 10cc. of absolute alcohol, mixed in a stoppered flask with 15cc. of ether and allowed to stand until clear, when the clear liquid is poured off into a glass stoppered weighing glass, filtering the last portions of the solution. The solution is then evaporated in the weighing glass until the residue no longer flows readily, after which it is dried an hour longer in a water jacket. After cooling it is weighed.

In the case of sweet wines (over 0.5 grams sugar in 100cc.) 50cc. are taken in a good sized flask, some sand added, and a sufficient quantity of powdered slack-lime, and heated with frequent shaking in the water bath. After cooling, 100cc. of 96 per cent. alcohol are added, the precipitate which forms allowed to separate, the solution filtered, and the residue washed with alcohol of the same strength. The alcoholic solution is evaporated and the residue treated as above.

Free acids (total quantity of the acid reacting constituents of the wine).—These are to be estimated with a sufficiently dilute normal solution of alkali (at least one-third normal alkali) in 10 to 20cc. wine. If one-tenth normal alkali is used at least 10cc. of wine should be taken for titration; if one-third normal, 20cc. of wine. The drop method (*Tupfel methode*), with delicate reagent paper, is recommended for the establishment of the neutral point. Any considerable quantities of carbonic acid in the wine are to be previously removed by shaking. These “free acids” are to be reckoned and reported as tartaric acid ($\text{C}_4\text{H}_6\text{O}_6$).

Volatile acids.—These are to be estimated by distillation in a current of steam, and not indirectly, and reported as acetic acid ($\text{C}_2\text{H}_4\text{O}_2$). The amount of the “fixed acids” is found by subtracting from the amount of “free acids” found, the amount of tartaric acid corresponding to the “volatile acids” found.

Bitartrate of potash and free tartaric acid.—(a) Qualitative detection of free tartaric acid: 20–30cc. of the wine are treated with precipitated and finely powdered bitartrate of potash, shaken repeatedly, filtered off after an hour, and 2–3 drops of a 20 per cent. solution of acetate of potash added to the clear filtrate, and the solution

allowed to stand twelve hours. The shaking and standing of the solution must take place at as nearly as possible the same temperature. If any considerable precipitate forms during this time free tartaric acid is present, and the estimation of it and of the bitartrate of potash may be necessary.

(b) Quantitative estimation of the bitartrate of potash and free tartaric acid: In two stoppered flasks two samples of 20cc. of wine each are treated with 200cc. ether-alcohol (equal volumes), after adding to the one flask 2-3 drops of a 20 per cent. solution of acetate of potash. The mixtures are well shaken, and allowed to stand 16-18 hours at a low temperature ($0-10^{\circ}\text{C.}$), the precipitate filtered off, washed with ether-alcohol, and titrated. (The solution of acetate of potash must be neutral or acid. The addition of too much acetate may cause the retention of some bitartrate in solution.) It is best on the score of safety to add to the filtrate from the estimation of the total tartaric acid a further portion of 2 drops of acetate of potash to see if a further precipitation takes place.

In special cases the following procedure of Nessler and Barth may be used as a control:

Fifty cubic centimeters of wine are evaporated to the consistency of a thin sirup (best with the addition of quartz sand), the residue brought into a flask by means of small washings of 95 per cent. alcohol, and with continual shaking more alcohol is gradually added, until the entire quantity of alcohol is about 100cc. The flask and contents are corked and allowed to stand four hours in a cool place, then filtered, and the precipitate washed with 95 per cent. alcohol; the filter paper, together with the partly flocculent, partly crystalline, precipitate, is returned to the flask, treated with 30cc. warm water, titrated after cooling, and the acidity reckoned as bitartrate. The result is sometimes too high if pectinous bodies separate out in small lumps, inclosing a small portion of free acids.

In the alcoholic filtrate the alcohol is evaporated, 0.5cc. of a 20 per cent. potassic acetate solution added, which has been acidified by a slight excess of acetic acid, and thus the formation of bitartrate from the free tartaric acid in the wine facilitated. The whole is now, like the first residue of evaporation, treated with (sand and) 95 per cent. alcohol, and carefully brought into a flask, the volume of alcohol increased to 100cc., well shaken, corked, allowed to stand in a cold place four hours, filtered, the precipitate washed, dissolved in warm water, titrated, and for one equivalent of alkali two equivalents of tartaric acid are reckoned.

This method for the estimation of the free tartaric acid has the advantage over the former of being free from all errors of estimation by difference. The presence of considerable quantities of sulphates impairs the accuracy of the method.

Malic acid, succinic acid, citric acid.—Methods for the separation and estimation of these acids cannot be recommenced at the present time.

Salicylic acid.—For the detection of this, 100cc. of wine are repeatedly shaken out with chloroform, the latter evaporated and the aqueous solution of the residue tested with very dilute solution of ferric chloride. For the approximately quantitative determination it is sufficient to weigh the chloroform residue, after it has been again recrystallized from chloroform.

Coloring matter.—Red wines are always to be tested for coal tar colors. Conclusions in regard to the presence of other foreign coloring matters drawn from the color of precipitates and other color reactions are only exceptionally to be regarded as safe. In the search for coal tar colors the shaking out of 100cc. of the wine with ether before and after its neutralization with ammonia is recommended. The ethereal solutions are to be tested separately.

Tannin.—In case a quantitative determination of tannin (or tannin and coloring matter) appears necessary the permanganate method of Neubauer is to be employed. As a rule the following estimation of the amount of tannin will suffice: The free acids are neutralized to within 0.5 grams in 100cc. with standard alkali, if necessary. Then 1cc. of 40 per cent. sodic acetate solution is added, and drop by drop a 10 per cent. solution of ferric chloride, avoiding an excess. One drop of ferric chloride is

sufficient for the precipitation of 0.05 per cent. of tannin. (New wines are deprived of the carbonic acid held in solution by repeated shaking.)

Sugar.—The sugar should be determined after the addition of carbonate of soda by means of Fehling's solution, using dilute solutions, and, in wines rich in sugar (*i. e.* wines containing over .5 gram in 100cc.), with observance of Soxhlet's modifications, and calculated as grape sugar. Highly colored wines are to be decolorized with animal charcoal if their content of sugar is low, and with acetate of lead and sodium carbonate if it is high.

If the polarization indicates the presence of cane sugar (compare under polarization) the estimation is to be repeated in the manner indicated after the inversion (heating with hydrochloric acid) of the solution. From the difference the cane sugar can be calculated.

Polarization.—(1) With white wines: 60cc. of wine are treated with 3cc. acetate of lead solution in a graduated cylinder, and the precipitate filtered off. To 30cc. of the filtrate is added 1.5cc. of a saturated solution of sodic carbonate, filtered again, and the filtrate polarized. This gives a dilution of 10:11 which must be allowed for.

(2) With red wines: 60cc. wines are treated with 6cc. acetate of lead, and to 30cc. of the filtrate 3cc. of the saturated solution of sodic carbonate added, filtered again, and polarized. In this way a dilution of 5:6 is obtained.

The above conditions are so arranged (with white and red wines) that the last filtrate suffices to fill the 220mm. tube of the Wild polaristrobometer of which the capacity is about 28cc.

In place of the acetate of lead very small quantities of animal charcoal can be used. In this case an addition of sodic carbonate is not necessary, nor is the volume of the wine altered. If a portion of the undiluted wine 220mm. long shows a higher right-handed rotation than 0.3° , Wild, the following procedure is necessary:

Two hundred and ten cubic centimeters of the wine are evaporated on the water bath to a thin sirup, after the addition of a few drops of a 20 per cent. solution of acetate of potash. To the residue is added gradually, with continual stirring, 200cc. of 90 per cent. alcohol. The alcoholic solution, when perfectly clear, is poured off or filtered into a flask, and the alcohol distilled or evaporated off down to about 5cc. The residue is treated with about 15cc. water and a little bone black, filtered into a graduated cylinder, and washed with water until the filtrate measures 30cc. If this shows on polarization a rotation of more than $+0.5^\circ$, Wild, the wine contains the unfermentable matter of commercial potato sugar (amylin). If in the estimation of the sugar by Fehling's solution more than 0.3 grams sugar in 100cc. was found, the original right-rotation caused by the amylin may be diminished by the left-rotating sugar; the above precipitation with alcohol is in this case to be undertaken, even when the right-rotation is less than 0.3° , Wild. The sugar is, however, first fermented by the addition of pure yeast. With very considerable content in (Fehling's solution) reducing sugar and proportionally small left-rotation, the diminishing of the left-rotation may be brought about by cane sugar or dextrin or amylin. For the detection of the first the wine is inverted by heating with hydrochloric acid (to 50cc. wine, 5cc. dilute hydrochloric acid of specific gravity 1.10), and again polarized. If the left-rotation has increased, the presence of cane sugar is demonstrated. The presence of dextrin is shown as given in the section on "gum." In case cane sugar is present well washed yeast, as pure as possible, should be added, and the wine polarized after fermentation is complete. The conclusions are then the same as with the wines poor in sugar.

For polarization only large, exact instruments are to be used.

The rotation is to be calculated in degrees Wild according to Landolt (*Zeitschr. f. analyt. Chemie*, 7. 9):

1° Wild	= 4.6043° Soleil.
1° Soleil	= 0.217189° Wild.
1° Wild	= 2.89005° Ventzke.
1° Ventzke	= 0.346015° Wild,

Gum (arabic).—For establishing the addition of any considerable quantities of gum 4cc. wine are treated with 10cc. of 96 per cent. alcohol. If gum is present, the mixture becomes milky, and only clears up again after several hours. The precipitate which occurs adheres partly to the sides of the tube, and forms hard lumps. In genuine wine flakes appear after a short time, which soon settle, and remain somewhat loose. For a more exact test it is recommended to evaporate the wine to the consistency of a sirup, extract with alcohol of the strength given above, and dissolve the insoluble residue in water. This solution is treated with some hydrochloric acid (of specific gravity 1.10) heated under pressure two hours, and the reducing power ascertained with Fehling's solution, and calculated to dextrose. In genuine wines no considerable reduction is obtained in this way. (Dextrin is to be detected in the same way.)

Mannite.—As the presence of mannite in wines has been observed in a few cases, it should be considered when pointed crystals make their appearance in the extract or the glycerine.

Nitrogen.—In the estimation of nitrogen the soda-lime method is to be used.

Mineral matters.—For their estimation 50cc. of wine are used. If the incineration is incomplete, the charcoal is leached with some water, and burned by itself. The solution is evaporated in the same dish, and the entire ash gently ignited.

Chlorine estimation.—The wine is saturated with sodic carbonate, evaporated, the residue gently ignited and exhausted with water. In this solution the chlorine is to be estimated volumetrically according to Volhard, or gravimetrically. Wines whose ashes do not burn white by gentle ignition usually contain considerable quantities of chlorine (salt).

Sulphuric acid.—This is to be estimated directly in the wine by the addition of barium chloride. The quantitative estimation of the sulphuric acid is to be carried out only in cases where the qualitative test indicates the presence of abnormally large quantities. (In the case of viscous or very muddy wines a previous clarification with Spanish-earth is to be recommended.)

If in a special case it is necessary to investigate whether free sulphuric acid or potassium bisulphate are present, it must be proved that more sulphuric acid is present than is necessary to form neutral salts with all the bases.

Phosphoric acid.—In the case of wines whose ashes do not react strongly alkaline the estimation is made by evaporating the wine with sodic carbonate and potassic nitrate, the residue gently ignited and taken up with dilute nitric acid; then the molybdenum method is to be used. If the ash reacts strongly alkaline the nitric-acid solution of it can be used directly for the phosphoric-acid determination.

The other mineral constituents of wine (also alum) are to be determined in the ash or residue of incineration.

Sulphurous acid.—One hundred cubic centimeters wine are distilled in a current of carbonic acid gas after the addition of phosphoric acid. For receiving the distillate 5cc. of normal iodine solution are used. After the first third has distilled off, the distillate, which must still contain an excess of free iodine, is acidified with hydrochloric acid, heated and treated with barium chloride.

Adulteration of grape wine with fruit wine.—The detection of this adulteration can only exceptionally be carried out with certainty by means of the methods that have so far been offered. Especially are all methods untrustworthy which rely upon a single reaction to distinguish grape from fruit wine; neither is it always possible to decide with certainty from the absence of tartaric acid or from the presence of only very small quantities that a wine is not made from grapes.

In the manufacture of artificial wine together with water the following articles are known to be sometimes used: Alcohol (direct or in the shape of fortified wine), cane sugar, starch sugar, and substances rich in sugar (honey), glycerine, bitartate of potash, tartaric acid, other vegetable acids, and substances rich in such acids, salicylic acid, mineral matters, gum arabic, tannic acid, and substances rich in the same (*e. g.*, kino, catechu), foreign coloring matters, various ethers and aromas.

The estimation or rather the means of detecting the most of these substances has already been given above with the exception of the aromas and ethers, for which no method can as yet be recommended.

The following substances may be mentioned here in particular, which serve for increasing the sugar, extract, and free acid: Dried fruit, tamarinds, St. John's bread, dates, figs.

B.—Rules for judging of the purity of wine.

I. (a) Tests and determinations which are, as a rule, to be performed in judging of the purity of wines: Extract, alcohol, sugar, free acids as a whole, free tartaric acid qualitative, sulphuric acid, total ash, polarization, gum, foreign coloring matters in red wines. (b) Tests and determinations which are also to be carried out under special circumstances: Specific gravity, volatile acids, bitartate of potash, and free tartaric acid quantitative, succinic acid, malic acid, citric acid, salicylic acid, sulphurous acid, tannin, mannite, special ash constituents, nitrogen.

The Commission considers it desirable, in giving the estimations generally performed, to adhere to the order of succession given above (under (a)).

II. The Commission cannot regard it as their province to give a guide for judging of the purity of wine, but thinks it advisable, in the light of its experience, to call attention to the following points:

Wines which are made wholly from pure grape juice very seldom contain a less quantity of extract than 1.5 grams in 100cc. wine. If wines poorer in extract occur they should be condemned, unless it can be proven that natural wines of the same district and vintage occur with a similar low content of extract.

After subtracting the "fixed acids" the remaining extract (*extractrest*) in pure wines, according to previous experience, amounts to at least 1.1 grams in 100cc., and after subtracting the "free acids," at least 1.0 gram. Wines which show less *extractrest* are to be condemned, in case it cannot be shown that natural wines of the same district and vintage contain as small an *extractrest*.

A wine which contains appreciably more ash than 10 per cent. of its extract content must contain, correspondingly, more extract than would otherwise be accepted as a minimum limit. In natural wines the relation of ash to extract approaches very closely 1 to 10 parts by weight. Still a considerable deviation from this relation does not entirely justify the conclusion that the wine is adulterated.

The amount of free tartaric acid in pure wines, according to previous experience, does not exceed one-sixth of the entire "fixed acids."

The relation between alcohol and glycerine can vary in pure wines between 100 parts by weight of alcohol to 7 parts by weight of glycerine; and 100 parts by weight of alcohol to 14 parts by weight of glycerine. In case of wines showing a different glycerine relation an addition of alcohol or glycerine can be inferred.

As sometimes during its handling in cellars small quantities of alcohol (at most 1 per cent. by volume) may find their way into wine this fact must be borne in mind in judging of its purity.

These proportions are not always applicable to sweet wines.

For the individual ash constituents no generally applicable limits can be given. The opinion that the better kinds of wine always contain more phosphoric acid than others is unfounded.

Wines that contain less than 0.14 gram of mineral matter in 100cc. are to be condemned, if it cannot be shown that natural wines of the same kind and the same vintage, which have been subjected to like treatment, have an equally small content of mineral matter.

Wines which contain more than 0.05 gram of salt in 100cc. are to be condemned.

Wines that contain more than 0.092 gram sulphuric acid (SO_3) corresponding to 0.20 grams potassic sulphate (K_2SO_4) in 100cc., are to be designated as wines containing too much sulphuric acid, either from the use of gypsum or in some other way.

Through various causes wines may become viscous, black, brown, cloudy, or bitter; they may otherwise change essentially in color, taste, and odor. The color of red wines may also separate in a solid form; still all these phenomena in and of themselves would not justify the condemnation of the wine as not genuine.

If during the summer time an energetic fermentation commences in a wine, this does not justify the conclusion that an addition of sugar or substances rich in sugar, *e. g.*, honey, &c., has taken place, for the first fermentation may have been hindered in various ways or the wine may have had an addition of a wine rich in sugar.

The methods adopted by the "Union of Bavarian Chemists" differ considerably from the above in many particulars, so they are given also, together with the methods adopted by the same body for the examination of beer¹ in somewhat condensed form.

WINES.

METHODS OF INVESTIGATION.

I. *Determination of specific gravity.*—This is to be done by means of a Westphal's balance or a pycnometer, and always at 15° C.

II. *Determination of extract.*—Ten to 50cc. wine at 15° C. are evaporated in a platinum dish on the water bath to the proper consistence and then dried in a drying oven at 100° C. to constant loss of weight. Constant loss of weight is assumed when three weighings, with equal intervals between the first and second and second and third give equal differences between the successive weighings.

Weighings are to be made at intervals of fifteen minutes.

III. *Inorganic matter.*—This is the incombustible ash obtained by burning the extract. Repeated moistening, drying, and heating to redness are advisable to entirely get rid of all organic constituents.

IV. *Acidity.*—After shaking vigorously, to drive off carbonic acid, the wine is to be titrated with an alkali solution and the acidity expressed in terms of tartaric acid.

V. *Glycerine.*—(1) This is determined in dry wines as follows: The alcohol is driven off from 100cc. wine, lime or magnesia added, and the mass evaporated to dryness. The residue is boiled with 90 per cent. alcohol, filtered, and the filtrate evaporated to dryness. This residue is dissolved in 10–20cc. alcohol, 15–30cc. ether added, and the mixture allowed to stand until it is clear. It is then decanted from the sticky precipitate into a glass-stoppered weighing bottle, evaporated to constant loss of weight, and weighed.

(2) The following method is employed for sweet wines: 100cc. wine are measured into a porcelain dish and evaporated on the water bath to a sirupy consistence, mixed with 100–150cc. absolute alcohol, poured into a flask, ether added in the proportion of 1½ volumes to each volume of alcohol used, the flask well shaken, and allowed to stand until the liquid becomes clear. This is then poured off and the residue again treated with a mixture of alcohol and ether. The liquids are mixed, the alcohol and ether driven off, the residue dissolved in water, and treated as in (1).

(3) In all glycerine determinations it is necessary to take into consideration the loss of glycerine due to its volatility with water and alcohol vapor, and accordingly to add to the glycerine found 0.100 gram for each 100cc. of liquid evaporated.

(4) It is necessary to test the glycerine from sweet wines for sugar, and if any is present it must be estimated by Soxhlet's or Kuapp's method and its weight subtracted from that of the glycerine.

VI. *Alcohol.*—The determination must be made by distillation in glass vessels and the results stated as follows: 100cc. wine at 15° C. contain x grams or cubic centimeters alcohol.

VII. *Polarization.*—(1) The wine is decolorized with plumbic subacetate.

¹ Hilger, Vereinbarungen u. s. w., p. 154.

(2) A slight excess of sodic carbonate is added to the filtrate from (1). Two cubic centimeters of a solution of plumbic subacetate are added to 40cc. white wine and 5cc. to 40cc. red wine, the solution is filtered and 1cc. of a saturated solution of sodic carbonate added to 21 or 22.5cc. of the filtrate.

(3) The kind of apparatus used and the length of the tube are to be given, and results estimated in equivalents of Wild's polaristrobometer with 200mm. tubes.

(4) All samples rotating more than 0.5° to the right (in 220mm. tubes, after treating as above), and showing no change, or but little change, in their rotatory power after inversion, are to be considered as containing unfermented glucose (starch sugar) residue.

(5) Rotatory power of less than 0.3° to the right shows that impure glucose has not been added.

(6) Wines rotating between 0.3° and 0.5° to the right must be treated by the alcohol method.

(7) Wines rotating strongly to the left must be fermented and their optical properties then examined.

VIII. *Sugar*.—This is to be determined by Soxhlet's or Knapp's method. The presence of unfermented cane sugar is to be shown by inversion, &c.

IX. *Potassic bitartrate*.—The determination of potassic bitartrate as such is to be omitted.

X. *Tartaric, malic, and succinic acids*.—(1) According to Schmidt and Hiepe's method.

(2) Determination of tartaric acid according to the modified Berthelot-Fleury method.

(3) If the addition of 1 gram finely powdered tartaric acid to 100 grams wine produces no precipitate of potassic bitartrate, the modified Berthelot-Fleury method must be employed to determine free tartaric acid.

XI. *Coloring matter*.—(1) Only aniline dyes are to be looked for.

(2) Special attention is to be paid to the spectroscopic behavior of rosaniline dyes, as obtained by shaking wines with amyl alcohol before and after saturation with ammonia.

(3) A qualitative test for alumina is not sufficient evidence of the addition of alum.

XII. *Nitrogen*.—To be determined according to the ordinary method.

XIII. *Citric acid*.—Presence to be shown by a qualitative test, as baric citrate.

XIV. *Sulphuric acid*.—To be determined in the wine after adding hydrochloric acid.

XV. *Chlorine*.—To be determined in the nitric-acid solution of the burnt residue by Volhard's method.

XVI. *Lime, magnesia, and phosphoric acid*.—These are determined in the ash fused with sodic hydrate and potassic nitrate, the phosphoric acid by the molybdenum method.

XVII. *Potash*.—Either in the wine ash, as the platinum double salt, or in the wine itself, by Kayser's method.

XVIII. *Gums*.—Presence shown by precipitation by alcohol; 4cc. wine and 10cc. 96 per cent. alcohol are mixed. If gum arabic has been added, a lumpy, thick, stringy precipitate is produced; whereas pure wine becomes at first opalescent and then flocculent.

METHODS OF JUDGING PURITY—(Beurtheilung).

Part I.

1. Commercial wines may be defined as follows: (a) The product obtained by the fermentation of grape juice with or without grape skins and stems. (b) The product obtained by the fermentation of pure must, to which pure sugar, water, or infusion of grape skins has been added. It must contain not more than 9 per cent. alcohol and 0.3 per cent. sugar, and not less than 0.7 per cent. acid, estimated as tar-

taric. (c) The product obtained in southern countries by the addition of alcohol to fermented or partly fermented grape juice. French wines are not included, however. (d) The product obtained by fermenting the expressed juice of more or less completely dried wine grapes.

II. The above definitions do not apply to champagnes.

III. The following include the operations undergone by wines in cellars (*Keller-mässige Behandlung*): (a) Drawing and filling. (b) Filtration. (c) Clarification by the use of kaolin, isinglass, gelatine or albumin, with or without tannin. (d) Sulphuring. Only minute traces of sulphurous acid may be contained in wine for consumption. (e) Adulteration of wine. (f) Addition of alcohol to wine intended for export.

IV. Wines, even if plastered, must not contain more sulphuric acid than that corresponding to 2 grams potassic sulphate (K_2SO_4) per liter.

V. Medicinal wines are those mentioned in Parts I and IV, with the following restrictions: (a) They must not contain more sulphuric acid than corresponds to 1 gram potassic sulphate per liter. (b) They must contain no sulphurous acid. (c) The percentage of alcohol and sugar to be given on the label. (d) These restrictions apply only to wines expressly recommended or sold for medicinal use.

Part II.

I. Improperly gallized wines are preparations of grape juice, pure sugar and water, or grape-skin infusion, that contain more than 9 per cent. alcohol or less than 0.7 per cent. acid, or both, and preparations in which impure glucose has been used. The following facts enable us to detect them: Small quantity of inorganic matter (phosphoric acid and magnesia), and right rotation if impure glucose is used. If the rotation exceeds 0.2° to the right, the wine is to be concentrated, freed from tartaric acid as far as possible, and again polarized.

II. Addition of alcohol is to be assumed if the ratio of alcohol to glycerine is greater than 10 to 1 by weight.

III. Addition of water and alcohol is recognized by the diminution in the quantity of inorganic matter, especially magnesia, phosphoric acid, and usually potash. Addition of water alone is recognized in the same way.

IV. Scheelization, *i. e.*, addition of glycerine, is assumed if the ratio of glycerine to alcohol exceeds 1 to 6 by weight.

V. The presence of cane sugar is ascertained by a determination of sugar (by Soxhlet's or Knapp's method), before and after inversion.

BEER.

A.—METHODS OF INVESTIGATION.

By beer is to be understood a fermented and still fermenting drink, made from barley (or wheat) malt, hops, and water, and which was fermented by yeast.

I. *Determination of specific gravity.*—For this as well as all other determinations the beer is freed from carbonic acid, as far as possible, by half-filling bottles with it and shaking vigorously. It is then filtered. The specific gravity is then determined either by Westphal's balance or by a pycnometer at $15^\circ C$.

II. *Determination of extract.*—Seventy-five cubic centimeters of beer are carefully weighed and evaporated in a suitable vessel to 25cc., care being taken to prevent boiling. After cooling, water is added until the original weight is reached, and the specific gravity of the liquid taken as in I. The per cent. of extract is obtained from this specific gravity by the use of a table constructed by Dr. Schultz, and is given as "per cent. extract, Schultz."¹

¹Hilger, p. 123.

III. Alcohol is determined by distilling the beer. A picnometer of about 50cc. capacity and with a graduated neck is used as a receiver. The picnometer is carefully calibrated. Seventy-five cubic centimeters of beer are distilled until the distillate reaches about the center of the scale on the neck of the picnometer. This is then cooled to 15° C., dried, and weighed, and the alcohol determined by means of Baumber's table.

$$A = \frac{D \cdot d}{g}$$

The percentage of alcohol by weight is to be given. In very acid beers it is necessary to neutralize before distilling.

IV. *Original gravity of wort*.—This may be ascertained, approximately, by doubling the per cent. by weight of alcohol found as above, and adding the per cent. of extract. As this procedure is not exact, it may be made more nearly so by using the formula

$$\frac{100(E + 2.0665 A)}{100 + 1.0665 A}$$

V. Degree of fermentation. This is estimated by using the formula

$$V_1 = 100 \left(1 - \frac{E}{c} \right).$$

VI. *Sugar determination*.—This is to be determined directly, in the beer previously freed from carbonic acid, by Soxhlet's method of weighing the reduced copper; 1.13 parts of copper correspond to 1 part anhydrous maltose.

VII. Determination of dextrin is seldom required, and if required is to be performed by Sachsse's method.

VIII. *Nitrogen*.—Twenty to thirty cubic centimeters are evaporated in a Hofmeister "schälchen" or on warm mercury, and the extract burned with soda-lime. The nitrogen may also be determined by Kjeldahl's method.

IX. *Acids*.—(a) Total acids: The carbonic acid is driven off from 100cc. of beer by heating in beakers for a short time to 40° C. and the beer then titrated with baryta water (one-fifth to one-tenth normal). The saturation point is reached when a drop of the liquid has no longer any action on litmus paper. The acidity is to be given in cubic centimeters normal alkali required for 100 grams beer and as grams per cent. of lactic acid. The indication "acidity" or "degree of acidity" is insufficient.

(b) Normal beer contains but a very small quantity of acetic acid. The determination of fixed acid in the repeatedly evaporated extract is to be cast aside. The acetic acid produced by souring of the beer is shown by the increase in total acids. A qualitative test of the presence of acetic acid in the distillate from beers containing acetic acid is sufficient. Neutralized beer is to be acidified with phosphoric acid and distilled. Weigert's method is recommended.

X. *Ash*.—Thirty to fifty cubic centimeters of beer are evaporated in a large tared platinum dish and the extract carefully burned. If the burning takes place slowly, the ash constituents do not fuse together.

XI. *Phosphoric acid*.—This is to be determined in the ash obtained by evaporating and burning in a muffle 50 to 100cc. of beer to which not too much baric hydrate has been added. The phosphoric acid is determined in the nitric acid solution of the ash by the molybdenum method.

XII. *Sulphuric acid*.—The direct determination is not permissible. The determination is to be made by using the ash prepared by burning with sodic hydrate and potassic nitrate or baric hydrate and proceeding in the ordinary way.

XIII. *Chlorine*.—This is to be determined in the ash prepared with sodic hydrate.

XIV. *Glycerine*.—Three grams calcic hydrate are added to 50cc. of beer, evaporated to a sirupy consistence, about 10 grams coarse sea-sand or marble added, and dried. The dry mass is rubbed up, put into a capsule of filter paper, placed in an extraction apparatus, and extracted for six to eight hours with 50cc. alcohol. To the light colored extract at least an equal volume of ether is added, and the solution, after stand-

ing a while, poured into or filtered into a weighed capsule. After evaporating the alcohol and ether, the residue is heated in a drying oven at 100° to 105° C. to a constant loss of weight. In beers rich in extract the ash contained in the glycerine can be weighed and subtracted. In case the glycerine contains sugar, this can be determined by Soxhlet's method and subtracted.

XV. Hop substitutes are to be determined by Dragendorff's method. Picric acid is to be determined by Fleck's method. In examining for alkaloids, check experiments with pure beer must in all cases be made.

XVI. *Sulphites*.—One hundred cubic centimeters of beer are distilled after the addition of phosphoric acid and the distillate conducted into iodine solution. After one-third has distilled over, the iodine-colored distillate is acidified with hydrochloric acid and baric chloride added. If sulphites were not contained in the beer no precipitate is observed, but at the utmost a turbidity.

XVII. *Salicylic acid*.—This may be shown qualitatively by shaking with ether, chloroform, or benzine. The solution is allowed to evaporate, the residue dissolved in water, and a very dilute solution of ferric chloride added. The addition of too much acid and too violent shaking is to be avoided. The smallest trace of salicylic acid may also be shown by dialysis, as it passes very readily through membrane.

NOTE.—All the results of an investigation are to be stated in percentages by weight.

B.—METHODS OF JUDGING PURITY OF BEERS.

I. It is unjust to demand in a fermented beer an exact ratio of alcohol to extract, as the brewer cannot regulate the degree of fermentation within narrow limits. As a rule, Bavarian draft and lager beers contain from 1.5 to 2 parts of extract for each part of alcohol, but a smaller proportion of extract would not necessarily prove the addition of alcohol or glucose (the former to the beer, the latter to the wort).

II. The degree of fermentation of a beer must be such that at least 48 per cent. of the original extract has undergone fermentation.

III. If glucose or other bodies poor in nitrogen have been used in appreciable quantity as substitutes for malt, the nitrogen contents of the beer extract will fall below 0.65 per cent.

IV. The acidity of a beer should not be greater than 3cc. normal alkali to 100cc. beer. Acidity of less than 1.2cc. normal alkali to 100cc. beer indicates previous neutralization. If the acids are composed principally of lactic acid a larger quantity may be present.

V. The ash of normal beer is not above 0.3 grams to 100 grams beer.

VI. The amounts of phosphoric and sulphuric acids and chlorine in beer extract vary within such wide limits that their determination signifies nothing as to the purity of the beer.

VII. The amount of glycerine in pure beer is not greater than 0.25 gram to 100 grams beer.

VIII. The following methods of clarifying beer are legal: (a) Filtration. (b) Well-boiled hazel or beech shavings. (c) Isinglass.

IX. The following methods of preserving beer are legal: (a) Carbonic acid. (b) Pasteurizing. (c) Salicylic acid, this only for beers intended for export to countries where the use of salicylic acid is not forbidden by law.

NOTE.—The preceding methods are also to be used in the examination of imported beer.

C.—ADMINISTRATIVE NOTE.

It is absolutely necessary that the beer be preserved in well-corked green-glass bottles. Stone jugs and such vessels are not to be used.

The beer samples are to be protected from light and kept at a low temperature.

Care in making tests is, above all, necessary.

APPENDIX C.

The following bill, recently adopted in New York, constitutes the only instance I have been able to find of legislation directed specifically against the adulteration of any form of fermented liquors in this country. The manner in which such bills are drawn has considerable influence upon their efficiency in preventing and punishing the fraud against which they are directed, and while it is not exactly within the province of this publication to make suggestions as to the proper form for such legislation, I feel impelled to venture the assertion that the crudity of this bill affords little evidence that any chemist was consulted or concerned in its composition:

AN ACT to define pure wines, half wines, made wines, and adulterated wines, and to regulate the manufacture and sale of half wines and made wines and to prohibit the manufacture or sale of adulterated wines within the State of New York.

[Passed June 18, 1887, three-fifths being present.]

The people of the State of New York, represented in Senate and Assembly, do enact as follows :

§ 1. All liquors denominated as wine, containing alcohol, "except such as shall be produced by the natural fermentation of pure undried fruit juice," or compounded with distilled spirits, or by both methods, except as permitted by section two of this act, whether denominated as wine or by any other name whatsoever, in the nature of articles for use as beverages, or for compounding with other liquors intended for such use, and all compounds of the same with pure wine, and all preserved fruit juices compounded with substances not produced from undried fruit, in the character of, or intended for use as beverages, or for use in the fermentation or preparation of liquors intended for use as beverages, and all wines, imitation of wines or other beverages produced from fruit which shall contain any alum, baryta salts, caustic lime, carbonate of soda, carbonate of potash, carbonic acid, salts of lead, glycerine, salicylic* acid, or any other antiseptic, coloring matter, other than that produced from undried fruit, artificial flavoring, essence of ether, or any other foreign substance whatsoever which is injurious to health, shall be denominated as adulterated wine, and any person or persons who shall manufacture with the intent to sell, or shall sell, or offer to sell, any of such wine or beverages shall be guilty of a misdemeanor, and shall be punished by a fine of not less than two hundred dollars, or more than one thousand dollars, or imprisonment in the county jail for a term of not less than six months, or more than one year, or by both such fine and imprisonment in the discretion of the court, and shall be liable to a penalty of one dollar for each gallon thereof sold, offered for sale, or manufactured with intent to sell, and such wine or beverage shall be deemed a public nuisance and forfeited to the State, and shall be summarily seized

* So in original.

and destroyed by any health officer within whose jurisdiction the same shall be found, and the reasonable expense of such seizure and destruction shall be a county charge.

§ 2. For the purpose of this act the words "pure wine" shall be understood to mean the fermented juice of undried grapes or other undried fruits, provided, however, that the addition of pure sugar to perfect the wine, or the addition of pure distilled spirits to preserve it, not to exceed eight per centum of its volume, or the using of the necessary things to clarify and fine the wine, which are not injurious to health, shall not be construed as adulterations, but such pure wine shall contain at least seventy-five per centum of pure grape or other undried fruit juice.

§ 3. For the further purpose of this act, should any person or persons manufacture with the intent to sell, or sell or offer to sell, any wine which contains less than seventy-five per centum and more than fifty per centum of pure grape or other undried fruit juice, and is otherwise pure, such wine shall be known, branded, marked, labeled and sold as "half wine," and upon each and every package of such wine, which shall contain more than three gallons, there shall be stamped upon both ends of such package, in black printed letters, at least one inch high and of proper proportion, the words "half wine," and upon all packages which shall contain more than one quart and up to three gallons, there shall be stamped upon each of such packages, in plain, printed black letters at least one-half inch high, and of proper proportion, the words "half wine," and upon all packages or bottles of one quart or less, there shall be placed a label, securely pasted thereon, on which label the words "half wine" shall be plainly printed in black letters at least one-fourth of an inch high and of proper proportion. Should any number of such packages be inclosed in a larger package, as a box, barrel, case, or basket, such outside package shall also receive the stamp "half wine," the letters to be of the size according to the amount of such wine contained in such outside package: *Provided, further,* That any person or persons who shall sell, offer for sale, or manufacture with the intent to sell any wine which shall contain less than fifty per centum of pure grape or other undried fruit juice, and is otherwise pure, such wine shall be known, stamped, labeled, and sold as "made wine," and shall be stamped, marked, and labeled in the same manner as prescribed in this section, except the words shall be in this case "made wine."

§ 4. If any person or persons shall sell, or offer for sale, or manufacture with intent to sell any wine of the kind and character as described in the third section of this act, which shall not be stamped, marked, or labeled after the manner and mode therein prescribed, such person or persons shall be guilty of a misdemeanor, and shall be punished by a fine of not less than two hundred dollars, or more than one thousand dollars for each and every offense, or by imprisonment in the county jail, not less than three months, or more than one year, or by both fine and imprisonment in the discretion of the court, and in addition thereto shall be liable to a penalty of one-half dollar for each gallon thereof so sold, offered for sale, or manufactured with the intent to sell or offer for sale. All penalties imposed by this act may be recovered with costs of action by any person, in his own name, before any justice of the peace in the county where the offense was committed; where the amount does not exceed the jurisdiction of said justice, or when such action shall be brought in the city of New York, before any justice of the district or of the city court of said city; and such penalties may be recovered in the like manner in any court of record in the State, but on recovery by the plaintiff in such case for a sum less than fifty dollars, the plaintiff shall only be entitled to costs to an amount equal to the amount of such recovery. It shall be the duty of any district attorney in this State, and he is hereby required to prosecute or commence actions in the name of the people of this State, for the recovery of the penalties allowed herein, upon receiving proper information thereof, and in all actions brought by such district attorney, one-half of the penalty recovered shall belong to and be paid over to the person or persons giving the information upon which the action is brought, and the other half shall be paid to the

treasurer of the county in which said action is brought within thirty days from the time of its collection, and the said one-half shall be placed to the credit of the poor fund of the town or city in which the cause of action arose. All judgments recovered in pursuance of the provisions of this act, with the interest thereon, may be collected and enforced by the same means and in the same manner as a judgment rendered in an action to recover damages for a personal injury. Two or more penalties may be included in the same action.

§ 5. The provisions of this act shall not apply to medicated wines, such as are put up and sold for medical purposes only.

§ 6. This act shall take effect on September first, eighteen hundred and eighty-seven.

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U. S. DEPARTMENT OF AGRICULTURE.

DIVISION OF CHEMISTRY.

BULLETIN

No. 13.

FOODS

AND

FOOD ADULTERANTS.

INVESTIGATIONS MADE BY AUTHORITY OF
THE COMMISSIONER OF AGRICULTURE,
UNDER DIRECTION OF
THE CHEMIST.

PART FOURTH:
LARD AND LARD ADULTERATIONS.
BY
H. W. WILEY.

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PART 4.—LARD AND LARD ADULTERATIONS

U. S. DEPARTMENT OF AGRICULTURE,
DIVISION OF CHEMISTRY,
Washington, D. C., February 7, 1889.

SIR: With many interruptions, due to the experiments in the manufacture of sugar, carried on under the supervision of this division, I have completed our studies on lard and lard adulterations, and now have the honor to lay before you the results obtained for your inspection and approval.

I have endeavored to show the character of true lard, how it is made, and how it may be distinguished from its imitations. In the same manner the substances used in adulterating lard—viz, stearines and cotton oil—have been studied and their properties described. Also the characteristics of the mixed lards have been pointed out, and the best methods of analytical research illustrated.

Abstracts of similar studies by others have been given, and it is believed that the present state of our knowledge of lard and its compounds is fully set forth.

Some delay in submitting the manuscript to the Public Printer has been experienced on account of failure to arrange for printing the illustrations. To facilitate this matter, it has been decided to omit nearly all illustrations of methods of making and refining lard and cotton oil, and print only a few photo-micrographs showing the crystalline appearance of pure lard and stearines and mixtures thereof.

Respectfully,

H. W. WILEY,
Chemist.

Hon. NORMAN J. COLMAN,
Commissioner of Agriculture.



LARD AND LARD ADULTERATIONS.

(1) LARD.

(a) *Lard* is a term applied to the fat of the slaughtered hog, separated from the other tissues of the animal by the aid of heat.

In the crude state it is composed chiefly of the glycerides of the fatty acids, oleic and stearic or palmitic, with small portions of the connective tissues, animal gelatine, and other organic matters.

(b) *Kinds of lard*.—According to the parts of the fat used and the methods of rendering it lard is divided into several classes. According to methods of rendering lard is classified as kettle and steam. From material used the following classification may be made:

(c) *Neutral lard*.—Neutral lard is composed of the fats derived from the leaf of the slaughtered animal, taken in a perfectly fresh state. The leaf is either chilled in a cold atmosphere or treated with cold water to remove the animal heat. It is then reduced to a pulp in a grinder and passed at once to the rendering kettle. The fat is rendered at a temperature 105° to 120° F. (40°–50° C.). Only a part of the lard is separated at this temperature and the rest is sent to other rendering tanks to be made into another kind of product. The lard obtained as above is washed in a melted state with water containing a trace of sodium carbonate, sodium chloride, or a dilute acid. The lard thus formed is almost neutral, containing not to exceed .25 per cent. free acid; but it may contain a considerable quantity of water and some salt. This neutral lard is used almost exclusively for making butterine (oleomargarine).

(d) *Leaf lard*.—The residue unrendered in the above process is subjected to steam heat under pressure and the fat thus obtained is called leaf lard. Formerly this was the only kind of lard recognized in the Chicago Board of Trade, and was then made of the whole leaf.

(e) *Choice kettle-rendered lard; Choice lard*.—The quantity of lard required for butterine does not include all of the leaf produced. The remaining portions of the leaf, together with the fat cut from the backs, are rendered in steam-jacketed open kettles and produce a choice variety of lard known as “kettle-rendered.” The hide is removed from

the back fat before rendering and both leaf and back fat are passed through a pulping machine before they enter the kettle. Choice lard is thus defined by the regulations of the Chicago Board of Trade:

Choice lard.—Choice lard to be made from leaf and trimmings only, either steam or kettle rendered, the manner of rendering to be branded on each tierce.

(f) *Prime steam lard.*—The prime steam lard of commerce is made as follows: The whole head of the hog, after the removal of the jawl, is used for rendering. The heads are placed in the bottom of the rendering tank. The fat is pulled off of the small intestines and also placed in the tank. Any fat that may be attached to the heart of the animal is also used. In houses where kettle-rendered lard is not made the back fat and trimmings are also used. When there is no demand for leaf lard the leaf is also put into the rendering tank with the other portions of the body mentioned. It is thus seen that prime steam lard may be taken to represent the fat of the whole animal, or only portions thereof. The quantity of fat afforded by each animal varies with the market to which the meat is to be sent. A hog trimmed for the domestic market will give an average of about 40 pounds, while from one destined for the English market only about 20 pounds of lard will be made. Prime steam lard is thus defined by the Chicago Board of Trade:

Prime steam lard.—Standard prime steam lard shall be solely the product of the trimmings and other fat parts of hogs, rendered in tanks by the direct application of steam, and without subsequent change in grain or character by the use of agitators or other machinery, except as such change may unavoidably come from transportation. It shall have proper color, flavor, and soundness for keeping, and no material which has been salted shall be included. The name and location of the renderer and the grade of the lard shall be plainly branded on each package at the time of packing.

This lard is passed solely on inspection; the inspector having no authority to supervise rendering establishments in order to secure a proper control of the kettles. According to the printed regulations, any part of the hog containing fat can be legally used.

Since much uncertainty exists in regard to the disposition which is made of the guts of the hog I have had the subject carefully investigated. Following are the results of the study:

(g) *Guts.*—The definition of the term as used by hog packers is: Everything inside of a hog except the lungs and hearts, or, in other words, the abdominal viscera complete. The material is handled as follows:

When the hog is split open the viscera are separated by cutting out the portion of flesh surrounding the anus and taking a strip containing the external urino-generative organs. The whole viscera are thrown on a table and divided as follows: The heart is thrown to one side and the fatty portion trimmed off for lard. The rest goes into the offal tank or sausage. The lungs and liver go into the offal tank (or sausage).

The rectum and large intestines are pulled from the intestinal fat and peritoneum and, along with the adhering flesh and genito-urinary organs, sent to the trimmer. All flesh and the above-mentioned organs are trimmed off and the intestine proper is used for sausage casings. The trimmings, including the genito urinary organs, are washed and dumped into the rendering tank. The small intestine is also pulled from the fatty membrane surrounding it and saved for sausage casings. The remaining material, consisting of the peritoneum, diaphragm, stomach, and adhering membranes, together with the intestinal fat, constitute the "guts" which are seen undergoing the process of washing, which is usually conducted in three or four different tanks. As the "guts" pass into the first tank the stomach and peritoneum are split open and also any portion of the intestines which sometimes adhere to the peritoneum. After receiving a rough wash they are passed from tank to tank, when, after the third or fourth wash, they are ready for the rendering tank. The omentum fat is cut from the kidneys and the kidneys with a little adhering fat go into the rendering tank. Spleen and pancreas go into the rendering tanks, as do also the trachea, vocal chords, and œsophagus.

To sum up, it is safe to say that everything goes into the rendering tank, with the following exceptions:

- (1) The intestines proper, which are saved for sausage-casings.
- (2) The liver and lungs.
- (3) That part of the heart free from fat.

I have been told that in killing small hogs, and also when there is small demand for sausage-casings, it is frequently the practice to split the intestines, so as to save expense of pulling from the fat, and after washing, fat and all go into the tank. Of course it will often happen that the intestines break off and portions adhere to the enveloping tissue, and consequently get into the tank after washing.

It is a commercial fact that sausage-casings are worth more than the small amount of adhering fat, and consequently packers will save them. Small hogs produce small casings difficult to pull, and it is reasonable to believe that they will be handled in the simpler manner. They break so easily that they are hardly worth saving separately. It is stated by lard manufacturers that the grease made from the parts of the intestines mentioned above is used for the manufacture of lard oil and soap, and does not enter into the lard of commerce.

(h) *Butchers' lard*.—The small quantities of lard made by butchers are usually "kettle-rendered," after the manner practiced by small farmers in making lard for home consumption. Often the scraps are saved up for a considerable length of time by the butchers before rendering, and that is likely to increase the free acid present. This lard is also frequently dark colored, and contains a considerable quantity of glue. In New York this lard is known as "New York City Lard."

In this figure is represented the type of apparatus used for rendering lard, etc., under pressure. The rendering vessel is made of boiler iron or steel, and varies in size according to the magnitude of the establish-

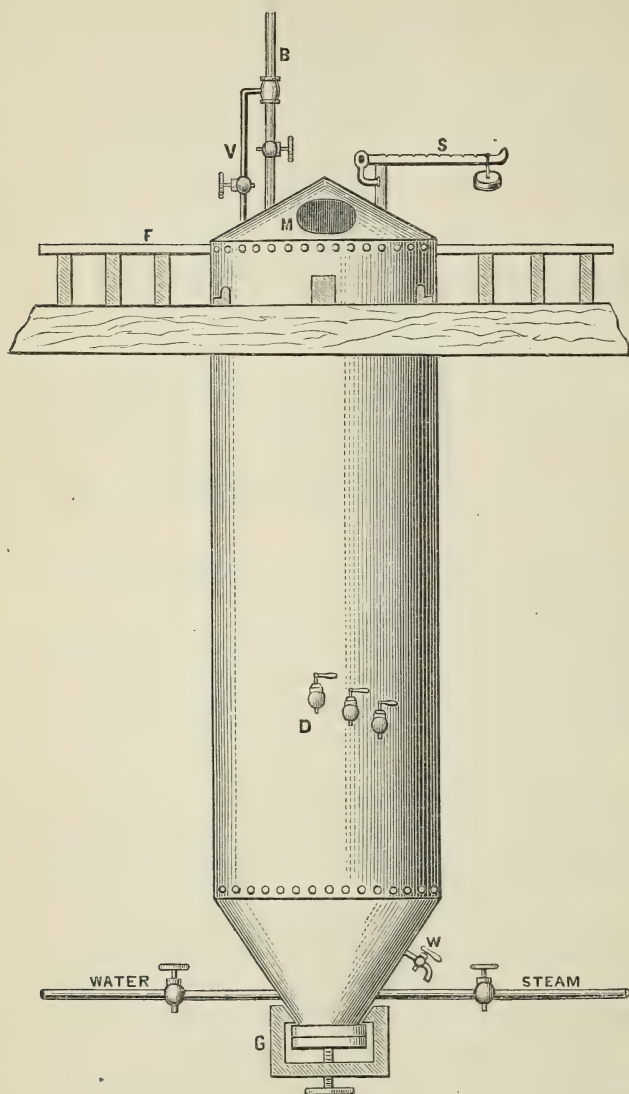


Fig. 16.

ment. A very common size is 10 to 12 feet in length and 3 to 5 feet in diameter. The heads, scraps, and other materials are put in at M. When the tank is full M is closed. Steam is admitted through the pipe thus marked, and condensed water drawn off through the water-pipe. Through the cocks at D the depth of lard in the tank can be determined

and the lard drawn off. When the process is finished and the lard drawn off the bottom G is opened and the "tankage" withdrawn and dried for fertilizing purposes.

(B) OTHER HOG-FAT PRODUCTS.

There are many other hog-fat products not used in the manufacture of lard or compound lard, a description of which, however, may prove useful here.

(a) *White grease*.—This grease is made chiefly from hogs which die in transit, by being smothered or frozen. Formerly it was also made from animals dead of disease; but this product has of late been diminished on account of certain State laws requiring the carcasses of hogs which have died of cholera to be buried. This grease is made from the whole animal with the exception of the intestines. The latter are rendered separately and make "brown grease". The rendering is done in closed tanks at a high pressure. The residue is used in the manufacture of fertilizer. White and brown grease are used chiefly in the manufacture of low-grade lard oils and soap.

(b) *Yellow grease*.—Yellow grease is made by packers. All the refuse materials of the packing-houses go into the yellow-grease tank, together with any hogs which may die on the packers' hands. Yellow grease is intermediate in value between white and brown. It is used for the same purposes.

(c) *Pigs' foot grease*.—This grease is obtained chiefly from the glue factories, and is used for making lard oils and soap.

(2) STEARINES.

The stearines are the more solid portions of the animal fats remaining after the more fluid portions have been removed by pressure. The stearines used in the manufacture of compound lard are lard stearine, derived from lard, and oleo stearine, derived from a certain quality of beef tallow. Cotton-oil stearine is used chiefly in the manufacture of butterine.

A.—LARD STEARINE.

The lard stearine used in compound lard is made as follows:

The prime steam lard, if properly crystallized and of the right temperature (from 45° to 55° F., winter; 55° to 65°, summer), is sent at once to the presses. If not properly grained, it is melted and kept in a crystallizing room at 50° to 60° F., until the proper grain is formed. The lard is then wrapped in cakes with cloth, each cake containing 10 to 20 pounds. The cakes are then placed in a large press, with suitable septa to facilitate the egress of the oil. These presses are sometimes 40 to 50 feet in length, and when first filled 12 to 18 feet high. The pressure is applied very gradually at first by means of a lever working a capstan, about which the chain is wrapped, attached to the upper movable part of the press.

The oil expressed, prime or extra lard oil, is used for illuminating and lubricating purposes. The resulting stearine is used for making compound lard and is worth more than the lard. It has about .5 per cent. free fatty acid (less than the lard oil), and crystallizes in long needles, making the texture tough.

B.—OLEO-STEARINE.

This product is made chiefly from the caul fat of beeves. This fat is rendered in open kettles at a low temperature. The resulting tallow is placed in cars in a granulating room, where it is allowed to remain for thirty-six to forty-eight hours at a temperature 80° to 90° Fah. The contents of the cars are then mixed and placed on a revolving table, where they are made into cakes. These are wrapped with strong cotton cloth and placed in a strong press, where a gradual pressure at 90° F., becoming very strong at the end, is applied for one or two hours. The expressed oil, known as oleo-oil, is used in the manufacture of butterine. The stearine is removed from the press as white hard cakes, and is used for adulterating lard. The oil is sometimes filtered with a small percentage of fuller's earth, to improve its color and brightness.

C.—MUTTON TALLOW.

A fine article of mutton tallow is also sometimes used in lard, but the objection to the flavor is sufficient to limit its use to a small amount.

D.—BEEF FAT.

The following general remarks on beef fat will be found instructive:

Before the day of the oleomargarine industry all fat rendered from the tissues of cattle was known commercially as tallow. Since then differentiation has taken place and the term tallow is no longer sufficient to designate the several products obtained from the rendered fat of the beef. We have first "butter stock," which is rendered from the caul fat at a low temperature and from which is manufactured by means of pressure—

(1) Oleo-oil.

(2) Oleo-stearine (beef stearine).

The kidney fat as a rule is left with the carcass and constitutes what is known as suet. Marrow stock, as its name implies, is rendered marrow fat, and when properly prepared is almost equal to butter stock in quality. Tallow is made from the trimmings and portions of the viscera. Its color varies from white to yellow according to the portions of the animal which have been used and the care with which they have been prepared for rendering and the temperature at which rendered. When freshly and carefully rendered tallow should show less than 1.5 per cent. of free fatty acid. The tallow on the market will show anywhere from 2 to 10 per cent. Its flavor varies, never being good enough for lard. Tallow grease corresponds to the yellow grease of the hog-packer. It is of a dark color and often contains as much as 50 per cent. of free acid. It is made into low-grade soaps.

(3) COTTON OIL.

(a) The cotton seed from various sources is put through a screen to take out the bolls and coarse material. The seed is then put through a gin to remove as far as possible any remaining lint, of which about 20 pounds per ton of seed are obtained.

The clean seed is next sent to a huller composed of revolving cylinders covered with knives, which cut up both seed and hull. The chips are then conveyed to a screen placed on a vibrating frame, through which the kernels fall. The hulls are carried by an endless belt to the furnaces, where they are burned. The kernels of the seed are conveyed to crusher rolls, where they are ground to a fine meal. The meal is then sent to a heater, where it remains from twenty to forty minutes. These heaters have a temperature of 210° to 215° F. The hot meal is formed into cakes by machinery; these are wrapped in cloth and placed in the press. About 16 pounds of meal are put in each cake. The cakes are placed in a hydraulic press, where a pressure of from 3,000 to 4,000 pounds per square inch is applied. The press is also kept warm. The expressed cakes contain only about 10 per cent. of oil. The cake is sold as cattle food or for fertilizing purposes. The crude oil as thus expressed contains about 1.5 per cent. of free acid. The chief cotton-seed presses of the country are located at the following points:

Cotton-seed oil milling points.

Arkansas:	Illinois:	North Carolina:
Little Rock.	Cairo.	Charlotte.
Argenta.	Louisiana:	Raleigh.
Fort Smith.	New Orleans.	Tennessee:
Texarkana.	Shreveport.	Memphis.
Brinkley.	Baton Rouge.	Jackson.
Helena.	Monroe.	Nashville.
Alabama:	Missouri:	Dyersburgh.
Selma.	Saint Louis.	Texas:
Mobile.	Mississippi:	Brenham.
Montgomery.	Clarksdale.	Dallas.
Eufaula.	Columbus.	Galveston.
Huntsville.	Canton.	Houston.
Georgia:	Grenada.	Palestine.
Atlanta.	Greenville.	Waco.
Augusta.	Meridian.	
Albany.	Natchez.	
Columbus.	Vicksburg.	
Macon.	West Point.	
Rome.		

The oil is chiefly pressed in winter, since it is difficult to keep the seed for summer work. Some mills are, however, operated during the summer. The crude oil is shipped in tanks holding from 36,000 to 45,000 pounds each. When the oil is shipped North in winter it usually becomes solidified. In order to get it out of the tanks they are placed on switches and a jet of steam is introduced into the tank and the oil gradually

melted out. Another method consists in covering the tank with wood, forming a chamber into which exhausted steam is introduced. Gutters are provided along the railroad tracks into which the oil flows and is conducted into the receiving tanks. From the receiving tanks it is pumped into large receivers called scale tanks, where the crude oil is weighed.

(b) *Refining process*.—After weighing, the oil is pumped into refining kettles. These are of various sizes, the largest ones being 20 to 25 feet deep and 15 feet in diameter. These tanks are furnished with steam-coils for the purpose of heating the oil and with appropriate machinery for keeping it in motion. A solution of caustic soda is used for refining. This solution is made from 10° to 28° Beaumé in strength, and varying quantities are used according to the nature of the oil operated upon. After the addition of the caustic soda the mixture is agitated for forty-five minutes and kept at a temperature of 100° to 110° F. The contents of the tank are then allowed to stand six to thirty-six hours, when the solid matters, soap and substances precipitated by the caustic alkali gather at the bottom. This mixture is called "foots," and is used for making soap. The yellow oil resulting by this process is further purified by being heated and allowed to settle again or by filtration and is called summer yellow oil. Winter yellow oil is made from the above material by chilling it until it partially crystallizes and separating the stearine formed, about 25 per cent., in presses similar to those used for lard. This cotton-oil stearine is used for making butterine and soap.

(c) *White oil*.—The yellow oil obtained as above is treated with from 2 to 3 per cent. of fuller's earth in a tank furnished with apparatus for keeping the mixture in motion. When the fuller's earth has been thus thoroughly mixed with the oil, the whole is sent to the filter press. The fuller's earth has the property of absorbing or holding back the yellow coloring matter, so that the oil which issues from the press is almost white. This white oil is the one which is chiefly used for making compound lard.

Cotton oil is obtained from the seeds of *Gossypium herbaceum*. The percentage of oil varies in the seed from 10 to 30.

In 1882 it was estimated that the oil industry was represented by the following data : *

410,000 tons of seed, yielding 35 gallons of crude oil to the ton, are	
14,350,000 gallons, worth 30 cents per gallon	\$4,305,000
Same amount of seed, yielding 22 pounds cotton lint to the ton, is 9,020,000	
pounds cotton, worth 8 cents per pound	721,600
And yielding also 750 pounds of oil-cake to the ton (2,240 pounds) is 137,277	
tons of cake at \$20 per ton	2,745,540
	<hr/>
	7,772,140
Deduct the sum paid for the seed, say	4,100,000
	<hr/>
And there remains for value gained in manipulation of seed	3,672,140

From September 1, 1883, to September 1, 1886, there were exported from New York 88,871 barrels, and from New Orleans 186,720 barrels, making a total of 275,591 barrels from the two ports. These figures show conclusively that American cotton-seed oil is growing rapidly in favor in foreign countries.

When well stored and properly ventilated, cotton seed keeps sweet for twelve months. If allowed to become damp, or stored too long in bulk, it grows heated, and is liable to spontaneous combustion.

Manufacture of cotton-seed oil.—The seed when landed at the mill is first examined. If too damp or wet it is dried by spreading it over a floor with free access of air, exposing it on frames to the sunlight in warm weather, or by kiln-drying. Drying is the exception rather than the rule in the United States. Cotton ginning is so carefully done that the seeds have little or no opportunity to become wet. Besides this, the seed is generally held at the gins for some time before it is sold to the oil manufacturer.

The first process in preparing the dry seed for the mill is to free it from dust. This is effected by shaking it in a screen or in drums lined with a fine metallic net and containing a strong magnet to which any iron nails will adhere, which are frequently present. From the drums the seeds drop into a gutter leading to a machine which removes the lint left by the gin. This is done by a gin constructed for the purpose, with saws closer together than the ordinary cotton-gin. An average of twenty-two pounds of short lint is taken from a ton of the seed. This product, called "linters," is used in the manufacture of cotton batting. The clean seeds are then transferred to the sheller, which consists of a revolving cylinder containing twenty-four cylindrical knives and four back knives. The sheller revolves at great speed, and as the seed is forced between the knives the pericarp or hull is broken and forced from the kernel. The mixed shells and kernels are separated in a winnowing machine by a strong blast of air. This removal of the husk makes a vast difference in the meal cake, a desiccated or decorticated cake being five times more nutritious and wholesome than an undecorticated cake.

Being thus cleaned, shelled, and separated, the kernels are carried by a system of elevators to the upper story and then pass down into the crusher-rolls to be ground to flour.

Cold pressure produces a very good salad oil, and this is the method generally pursued in Marseilles and other European cities for the first pressure, after which the residue is subjected to a second warm pressure. In this country, however, warm pressure is generally preferred. The meal is heated in a meal heater for fifteen to twenty minutes to 204.4° to 215.3° F.

The heated meal is placed in woolen bags, each holding sufficient seed for a cake. The bags are then placed between horse-hair mats backed with leather having a fluted surface inside to facilitate the escape of the oil under the hydraulic pressure amounting to 169 tons. With the most improved presses the hair mats are, however, done away with. The bags remain in the press seventeen minutes, the solid "oil-cake" of commerce remaining behind. This cake forms a superior feed for cattle, horses, sheep, and especially swine, and is nutritious, easily digested, and fattening.

Cotton-seed cake is of a rich golden color, quite dry, and has a sweet, nutty, oleaginous taste. When ground to the fineness of corn meal it is known as "cotton-seed meal," and in that form is frequently used for fertilizing purposes.

The crude oil as obtained from the press is pumped into the oil-room and either barreled for shipment or refined.

Four qualities of the oil are known :

Crude oil is thickly fluid and of a dirty yellow to reddish color; on standing it deposits a slimy sediment. The *second* quality has a pale orange color and is obtained by refining the crude oil. The *third* quality is obtained by further purification of the second; and the *fourth*, which has a pale straw color and a pure nutty taste, by bleaching the third quality.

The coloring principle, termed *gossypin*, is collected on a filter, carefully washed to remove any trace of acid, and dried slowly at a low temperature. It is then ready for use as a dye, and gives fast colors on both silk and wool. It is claimed that the quantity of coloring matter in a ton of crude oil is 15 pounds, though this proportion must vary considerably. Its properties are insolubility in acids, slight solubility in water, free solubility in alcohol or alkalies. In its dry state it is a light powder of a pungent odor, of a brown color, and strongly tinctorial.

Crude cotton-seed is thickly fluid, twenty-eight to thirty times less fluid than water, and has a specific gravity of 0.9283 at 68° F., 0.9306 at 59° F., and 0.9343 at 50° F.

According to the quality of the oil, palmitin is separated between 54° and 43° F. The oil congeals at 28.5° to 27° F. In taste and odor it resembles linseed oil, and as regards other properties it is an intermediate between drying and non-drying oils.

Refined cotton-seed oil has a specific gravity of 0.9264 at 59° F.; it separates palmitin already below 53.5° F., and congeals at 32° to 30° F.

The oil consists of palmitin and olein, and to make it still more adapted for the adulteration of olive oil, for which immense quantities are used every year, it is intentionally cooled for the separation of palmitin, which lowers the specific gravity.

MIXING.

The term refined lard has long been used to designate a lard composed chiefly of cotton oil and stearine. The largest manufacturers of this kind of lard have now abandoned this term and are using the label "lard compound" instead. This is but just to the consumers of this article who are likely to be misled by the term refined lard. The prime steam lard in a state of fusion, the stearine also in a liquid condition, and the refined cotton oil are measured in the proportions to be used and placed in a tank at a temperature of 120° to 160° F. In this tank the ingredients are thoroughly mixed by means of paddles operated by machinery. After mixing the compound lard passes at once to artificial coolers where it is chilled as soon as possible. It is thence run directly into small tin cans or large packages and prepared for market.

(4) PROPERTIES OF PURE LARD.

A.—PHYSICAL PROPERTIES.

(a) *Specific gravity*.—The specific gravity of a pure lard varies rapidly with the temperature. It is not convenient to take the specific gravity of a lard at a lower temperature than 35° or 40°,* inasmuch as below that temperature solidification is apt to begin. The specific gravity, therefore, is usually taken at 35° or 40° or at the temperature of boiling water, viz, 100°. At 40° the specific gravity of pure lard is about .890, and at 100° about .860, referred to water at 4°.

The specific gravity of pure lard does not differ greatly from that of many of the substances used in adulterating it, but it is distinctly lower than that of cotton oil, and is of great distinctive value in analysis.

(b) *Melting point*.—The melting point of a pure lard is a physical

* All degrees are Centigrade unless otherwise stated.

characteristic of great value. The melting point of the fat of the swine varies with the part of the body from which it is taken. The fat from the foot of the swine appears to have the least melting point, viz, 35.1°. The intestinal fat seems to have the highest, viz, 44°. In fat derived from the head of the animal the melting point is found to be 35.5°, while the kidney fat of the same animal shows a melting point of 42.5°. In steam lards, representing the lards passed by the Chicago Board of Trade, the melting point for ten samples was found to vary between 29.8° and 43.9°. In general it may be said that the melting point of steam lards is about 37° which is the mean of ten samples examined. In pure lards derived from other localities the melting point was also found to vary. A sample of lard from Deerfoot Farm, Southborough, Mass., was found to have a melting point of 44.9°, while a pure lard from Sperry & Barnes, New Haven, Conn., melted at 39°. The mean for eighteen samples was 40.7°. While the melting point can not be taken as a certain indication of the purity of a lard, nevertheless a wide variation from 40° in the melting point of a lard should lead at least to a suspicion of its genuineness, or that it was made from some special part of the animal. Perhaps one reason why the melting point has not been more highly regarded by analysts is because of the unsatisfactory method of determining it; but when it is ascertained by the method used in these investigations it becomes a characteristic of great value.

(c) *Color reaction.*—The coloration produced on pure lard by certain reagents serves as a valuable diagnostic sign in the analysis of lard and its adulterations. Various reagents have been employed for the production of characteristic colors in fats, but of these only two are of essential importance. They are sulphuric and nitric acids. Pure lard, when mixed with sulphuric and nitric acids of the proper density, as indicated hereafter, give only a slight color which varies from light pink to faint brown. The variation produced in the colors by pure lards is doubtless due to the presence in various quantities of certain tissues of the animal other than fat. For instance, a variation in the amount of gelatinous substance mechanically entangled with the lard or of the tissues composing the cells in which the lard was originally contained would be entirely sufficient to account for the slight differences in color produced by lards of known purity. It might, therefore, be difficult to distinguish accurately between a pure lard containing a considerable amount of other tissues from the animal and one which contained a small amount of adulteration. The coloration produced, therefore, by the acids named should not be relied upon wholly in distinguishing pure and adulterated lards; but the character of such coloration should be carefully noted in the analyst's book. In the steam lards examined some of the remarks describing the coloration produced are as follows:

"Trace of color," "faint pink," "bright pink," "light red," "yellowish," etc. For pure lards of miscellaneous origin some of the descrip-

tions are as follows: "Brownish pink," "trace of yellow," "marked red brown," "no color," "slight coloration, etc.

(d) *Refractive index*.—The deviation produced in the direction of a ray of light in passing through a film of melted fat is also a valuable physical characteristic. This deviation is usually measured as the quotient $n = \frac{\text{sine of the angle of incidence}}{\text{sine of the angle of refraction}}$ and is known as the refractive index. The refractive index of pure water, at 25° on the instrument used in these investigations was 1.3300. The refractive index of the samples of lard was made at as low a temperature as possible to preserve fluidity, viz: between 30° and 36°. In the tables the temperature at which the index was taken is not given, but the number representing the index corrected to the uniform temperature 25°. The rate of variation in the refractive index for each degree of temperature, experimentally determined, for lard oil was .000288. This number may also be taken to represent the variation for lard. The refractive index varies inversely as the temperature. The mean number for a pure lard at 25° is about 1.4620. The variation from this number can be seen in the analytical tables which follow. The refractive index of pure lard is distinctly less than that of cotton-seed oil at the same temperature, and is therefore a valuable characteristic for analytical purposes.

(e) *Rise of temperature with sulphuric acid*.—More valuable for diagnostic purposes than the physical properties already considered is the rise of temperature which lard undergoes when mixed, under proper conditions, with sulphuric acid. There is such a marked difference between the numbers representing the rise of temperature in pure lard and those of the adulterants usually employed in the manufacture of mixed lard as to give this number a high analytical value. With steam lards, ten samples, the extremes, as registered by the thermometer, were 38.8° and 42.1°. For pure lards of miscellaneous origin, one from Deerfoot Farm, Southborough, Mass., gave a rise of temperature 37.1°, and a pure leaf lard from Sperry & Barnes, New Haven, Conn., a rise of temperature of 46.2°.

The value of this characteristic is so great as to lead me to expect approximately reliable quantitative results from a general determination of the actual amount of heat produced in an appropriate calorimeter. I am at present attempting to devise an instrument by which the actual number of calories produced by mixing definite quantities of fats and oils and sulphuric acid can be accurately determined.

(f) *Crystallization point of fatty acids*.—The method described in the work of Dalican for determining the crystallizing points of fatty acids gives valuable data concerning the nature of pure lard, and also of the relative amount of stearic and oleic acids present in the mixture. The crystallizing point was found to vary in the ten samples of prime steam lard already mentioned from 35.4° to 39.5°. In pure lards of other kinds the variation was found to be from 32.1° to 42.7°.

(g) *Melting point of fatty acids.*—In connection with the crystallizing point of the fatty acids, the melting point is also of value. This temperature has been determined in the fat acids derived from steam and pure lards, and the numbers will be found in the analytical tables. In the prime steam lards these numbers vary from 41.4° to 43° . In pure lards of other kinds the variation was from 36.9° to 46.6° .

B.—CHEMICAL PROPERTIES.

(a) *Volatile acids.*—The quantity of volatile acid, as ordinarily estimated in a pure lard, is quite minute. Unless some suspicion of adulteration is awakened the search for such volatile or soluble acid may be omitted. Measured by the decinormal alkali solution required for 5 grammes of the fat the mean quantity of volatile acid in a pure lard may vary from .2 to .4 of a cubic centimeter. The determination, therefore, of the volatile acid in the examination of lards has none of that high diagnostic value which attaches to it in the examination of butters.

(b) *Fixed acids.*—The quantity of fixed acids (non-volatile and insoluble in water) in lard varies from 93 to 95 per cent.

(c) *Free acids.*—The quantity of free acids in lard rarely exceeds .5 per cent.

Twelve determinations of free acids in lards of known purity gave the following numbers expressed as per cent.:

.54 .92 .55 .75 .75 .35 .65 .60 .45 1.0 .40 .50

(d) *Saponification equivalent.*—The amount of caustic alkali necessary to saponify the fatty acids of the common glycerides is known as its saponification equivalent or number. The operation is usually known as Koettstoffer's process. The number of parts of a glyceride saponified by one equivalent of alkali is represented by one-third of the molecular weight of the glyceride in question. The saponification equivalent, therefore, represents the number of grams of an oil or fat saponified by one equivalent in grams of an alkali. The percentage of caustic potash used for saponifying a lard is about 20 and the mean saponification equivalent about 285. In the prime steam lards examined by us, the extreme variations were 276.14 and 290.05, and the mean 283.45. In pure lards of other kinds the extremes were 272.64 and 294.14, and the mean 280.33.

(e) *Iodine number* —The quantity of iodine absorbed by an oil or fat affords one of the most valuable indications of its constitution. The glycerides of the olein series have the property of absorbing the halogens. On the other hand the glycerides of the stearic series do not absorb iodine. Hence in a fat or oil from which the presence of linolein and its analogous bodies can be excluded the quantity of iodine absorbed may become a fairly accurate measure of the amount of oleic acid present. The lard derived from different portions of the swine varies largely in the amount of olein contained therein. For instance, a sample of intestinal lard absorbed 57.34 per cent. of iodine; the leaf lard from the

same animal absorbed 52.55 per cent., the foot lard 77.28 per cent., the head lard 85.03 per cent. In the prime steam lards mentioned the variation in the percentage of iodine absorbed was from 60.34 to 66.47 per cent., and the mean 62.86 per cent. In pure lards of other kinds the mean was 62.48 per cent. Thus in lards of known purity the amount of iodine absorbed will indicate the probable part of the animal from which the fat in the lard was derived. The wide variation between the iodine numbers of pure lard and those of the adulterants used in making compound lard serve to render this number of the greatest importance in analytical work.

(f) *The reaction with nitrate of silver.*—Pure lards, treated with a solution of nitrate of silver, after the method of Bechi, or the fatty acids thereof, after the method of Milliau, give no reduction of metallic silver, or, at most, only a trace and no or only a slight coloration. This fact is of the utmost importance in the analysis of lard.

(g) *Microscopical appearances.*—Lard, examined with the microscope, shows a definite crystalline structure, but does not plainly reveal the character of the crystals. When lard is slowly crystallized from ether, beautiful rhombic crystals of stearine are obtained, which are easily distinguished from the groups of fan-shaped crystals given by beef or mutton fat under similar conditions.

(h) *Moisture in lard.*—The quantity of water in pure lard varies from a mere trace to .7 per cent. Twelve determinations showed the following per cents.:

.7	.4	.2	.5	.6	.5
.2	.2	.2	.3	.3	.7

(5) PROPERTIES OF LARD ADULTERANTS.

COTTONSEED OIL.

A.—PHYSICAL PROPERTIES.

(a) *Specific gravity.*—Cottonseed oil being liquid at ordinary temperatures, its specific gravity can be easily taken at the temperature of the room. For purposes of comparison, the rate of variation in the specific gravity of the oil can be determined and its specific gravity at any given temperature calculated, or its specific gravity can be directly determined at 35°, 40°, or 100°, as may be desired, by comparison with water at the same temperature. In the samples examined the specific gravities of the oils at 35° vary from .9132 to .9154. The mean for nineteen samples is .9142. These numbers show the relative weight of the oil, an equal volume of water at the same temperature being taken as unity.

Specific gravity of refined cotton oil at different temperatures.

[Water at 15° C=1. Average, oil at 15°=.9218—at 100°.8683.]

Tempera- ture C°.	Specific gravity.	Weight cubic foot oil.	Tempera- ture C°.	Specific gravity.	Weight cubic foot oil.
		<i>Pounds.</i>			<i>Pounds.</i>
10	.9249	57.68	56	.8958	55.86
11	.9243	57.63	57	.8953	55.82
12	.9237	57.60	58	.8946	55.78
13	.9231	57.55	59	.8940	55.74
14	.9224	57.51	60	.8934	55.71
15	.9218	57.49	61	.8927	55.67
16	.9212	57.45	62	.8921	55.63
17	.9206	57.41	63	.8915	55.59
18	.9199	57.35	64	.8908	55.55
19	.9193	57.31	65	.8902	55.51
20	.9187	57.28	66	.8896	55.51
21	.9181	57.25	67	.8890	55.48
22	.9174	57.21	68	.8883	55.39
23	.9168	57.18	69	.8877	55.35
24	.9161	57.13	70	.8870	55.31
25	.9155	57.08	71	.8864	55.28
26	.9149	57.04	72	.8858	55.25
27	.9143	56.00	73	.8851	55.20
28	.9136	56.97	74	.8845	55.16
29	.9129	56.93	75	.8839	55.13
30	.9193	56.89	76	.8832	55.19
31	.9117	56.85	77	.8826	55.05
32	.9110	56.81	78	.8820	55.01
33	.9105	56.75	79	.8814	54.98
34	.9098	56.74	80	.8807	54.94
35	.9092	56.70	81	.8801	54.90
36	.9086	56.66	82	.8795	54.86
37	.9079	56.61	83	.8788	54.80
38	.9073	56.58	84	.8782	54.76
39	.9067	56.54	85	.8776	54.73
40	.9060	56.50	86	.8769	54.68
41	.9054	56.46	87	.8763	54.64
42	.9048	56.42	88	.8757	54.60
43	.9043	56.38	89	.8751	54.56
44	.9035	56.34	90	.8744	54.53
45	.9029	56.30	91	.8738	54.49
46	.9022	56.26	92	.8732	54.45
47	.9016	56.23	93	.8725	54.40
48	.9010	56.18	94	.8719	54.36
49	.9004	56.13	95	.8712	54.31
50	.8997	56.10	96	.8706	54.28
51	.8991	56.07	97	.8700	54.24
52	.8984	56.03	98	.8695	54.20
53	.8978	55.99	99	.8689	54.16
54	.8972	55.95	100	.8683	54.10
55	.8966	55.91			

(b) *Melting point*.—Since cotton oil solidifies only at a temperature near or below the freezing point of water its melting point has not been determined.

(c) *Color reaction*.—The color produced in cotton oil by sulphuric and nitric acids is a characteristic mark of the greatest value. This color varies from deep reddish brown to an almost black color. Some of the descriptions of the color produced in cotton oil, taken from the notebook, are as follows: "dark brown," "very brown black," "deep red brown," "very red," "yellow brown," etc. It must not be forgotten, however, that these colors can be produced by other oils, and hence their occurrence is not conclusive evidence of the presence of cotton oil.

(d) *Refractive index*.—The refractive index of cotton oil is distinctly higher than that of lard. The variation in the index of refraction is inversely as the temperature. The mean rate of variation for each degree is .000288. For a temperature of 25° the mean refractive index of the samples examined was 1.4674. The rate of variation in the index of refraction in cotton oil is sensibly the same as that for lard.

(e) *Rise of temperature with sulphuric acid*.—The rise of temperature which cotton oil suffers when mixed with sulphuric acid is a very prominent diagnostic sign. In the samples examined the lowest increment of temperature noted was 80.4° and the highest 90.2°. The mean rise of temperature was 85.4°. Cotton oil, therefore, gives more than double the increment of temperature shown by pure lard under the same conditions.

(f) *Crystallization point of fatty acids*.—Since cotton oil is fluid even at low temperatures (viz, 0°) the determination of its melting point is only a matter of scientific interest. The point at which its free acids crystallize is, however, easily determined according to the method of Dalican.

	Degrees.
The mean crystallizing point of the acids examined was.....	33.5
The minimum was	30.5
The maximum was.....	35.6

The high temperature reached in the crystallization of the fat acids is a peculiar characteristic of cotton oil. In lard there is not a very great difference between the temperatures indicated by the melting point of the glycerides and the crystallizing point of the fat acids. In cotton oil, however, these temperatures are widely removed.

(g) *Melting point of fatty acids*.—The melting point of the free acids of cotton oil was determined both in capillary tubes and by observing the deportment of the acid on the bulb of a delicate thermometer protected by a glass flask. The two sets of data were almost identical.

	Degrees.
The mean melting point of the acids examined was.....	39.1
Maximum	44.4
Minimum.....	34.6

The characteristics mentioned above are emphasized when the melting point of the fat acids is considered. These numbers seem much higher than would be expected.

B.—CHEMICAL PROPERTIES.

(a) *Volatile acids*.—The statements made in regard to the volatile acids in a pure lard are also applicable to cotton-seed oil.

For 5 grammes of cotton oil the quantity of deci-normal alkali consumed is slightly greater than for pure lard and may amount to as much as .5 cc.

If cocoa oil is present the number will be much higher. 5 grammes of pure cocoa oil will consume from .7 to .8 cc of the deci-normal alkali.

(b) *Saponification equivalent*.—In the samples reported the mean saponification equivalent was 283.8, although in some instances quite a difference was noticed from this figure.

(c) *Iodine number*.—Cotton oil possesses in a much higher degree than lard the property of absorbing iodine. This is due not only to the large percentage of oleic acid which it contains, but also probably to the presence of a small amount of linoleic acid or some homologue thereof. In the samples examined in no case did the iodine number fall below 100 and in one instance it rose to 116.97. The mean iodine number was 109.02.

(d) *Reaction with nitrate of silver*.—A more important property even than its power of absorbing iodine is shown by cotton oil in the reduction of silver to the metallic state under certain conditions. The test may be applied, as already indicated, either to the oil itself or to the fatty acids thereof. The silver is either reduced in the form of a metallic mirror deposited on the sides of the vessel or in minute black particles which give a brown or black appearance to the liquid. In some cases the liquid shows a greenish tint.

OTHER PROPERTIES.

The refined cotton oil used in adulterating lard has a pleasant taste, is almost odorless, and possesses a faint yellow color. Its resemblance to olive oil is so marked that for all culinary purposes it forms an excellent substitute therefor. Cotton oil possesses slight drying qualities which render it unfit for lubricating delicate machinery. Therefore it can never take the place of sweet oil for that purpose.

STEARINES.

The stearines used in the adulteration of lard are derived chiefly from lard, certain parts of beef fat, and cotton oil. These are generally called lard stearine, oleo-stearine, and cotton-oil stearine, respectively.

A.—PHYSICAL PROPERTIES.

(a) *Specific gravity*.—The specific gravity of stearines may be taken in their solid state or in a liquid state at a high temperature, 40° to 100° .

(b) *Melting point*.—The melting points of the stearines are higher than the natural glycerides from which they are derived. A prime oleo-stearine from Armour & Co., Chicago, showed a melting point of 51.9° . A prime lard stearine from the same firm showed a melting point of 44.3° , which is only slightly higher than the mean melting point of pure lards. The lowest melting point of any stearine examined was a sample of dead-hog stearine from J. P. Squire, Boston, which was 33.2° . The highest observed melting point in the stearines examined was an oleo-stearine from N. K. Fairbank & Co., Chicago, showing 53.8° . The high melting point of the stearines is a characteristic of great value in the adulteration of lard since it serves to counteract the influence of the cotton oil, which of course tends to lower the melting point of any lard mixture into which it may enter. The influence of the various constituents, however, on the melting point does not seem to be proportional to the respective quantity of each therein. For instance, a mixture of 25 per cent. of cotton oil having a melting point below zero, with 25 per cent. of an oleo-stearine having a melting point of only about 12° above the normal for pure lard, with 50 per cent. of pure lard of normal melting point, might not show a lowering of the melting point at all proportional to the presumable influence of the cotton oil present. The cotton-oil stearine, as might be expected, has a melting point below that of the similar products derived from lard and tallow.

(c) *Color reaction*.—The color reactions produced in the stearines by sulphuric and nitric acids are much the same as those produced in the original glycerides from which they were derived. Cotton-oil stearine shows a less intense color perhaps than the original oil; while in the case of tallow and lard stearines the coloration is not marked enough to be susceptible of description.

(d) *Refractive index*.—The refractive index of the stearines appears to be slightly lower than that of the original glycerides. The high refractive index which was noticed in the case of the original glycerides of the cotton-oil was also found in the stearine from that source.

(e) *Rise of temperature with sulphuric acid*.—With the lard and tallow stearines no degree of comparison can be made in the rise of temperature with that produced in the original glycerides, on account of the high initial temperature which is necessary for the conduct of the experiment. Allowing for the difference in initial temperature, however, the stearines depict themselves very much as the original glycerides.

B.—CHEMICAL PROPERTIES.

(a) *Volatile acids*.—The amount of volatile acids in the stearines mentioned is so small as to be negligible.

(b) *Saponification equivalent*.—The numbers are essentially the same as those of the original glycerides.

(c) *Iodine number*.—The percentage of iodine absorbed by the stearines is, as is to be expected from the fact that they contain less triolein, markedly less than that of the original glycerides. The fact that the stearines possess that property in this diminished degree is of quite as much importance from an analytical point of view as their high melting point. Thus the mixture of a stearine with a low iodine number with cotton oil of a high iodine number shows a percentage of iodine absorption not greatly different from that of pure lard. One prime oleo-stearine examined showed an iodine absorption of only 17.38 per cent. Another oleo-stearine showed 26.81 per cent. The lard stearines showed higher numbers, viz, in two cases 44.24 per cent. and 49.78 per cent. The cotton-oil stearines showed iodine numbers varying from 85.28 per cent. to 99.39 per cent.

(d) *Reaction with nitrate of silver*.—The stearines react with nitrate of silver in a manner entirely comparable with that of their original glycerides. The colors, however, are not so marked nor the precipitate of silver quite so abundant with cotton-oil stearines as with the oils themselves.

(e) *Microscopical appearances*.—Stearine derived from beef or mutton tallow shows under the microscope the characteristic fan shaped crystals already noticed. Lard stearine, on the other hand, gives crystalline groups similar to those already mentioned in the case of lard.

(f) *Moisture*.—Properly prepared stearine contains only a trace of moisture.

OTHER ADULTERANTS OF LARD.

It has been claimed that other substances than those mentioned have been used in the adulteration of lard, but these claims seem to rest on no valid foundation. Among these substances, dead-hog grease or dead-hog stearine is the one most frequently mentioned. The term dead-hog grease is used to indicate the oil or lard obtained from animals which die of disease, or are smothered in transportation, or die on the way to the slaughtering houses. The fat of animals very recently dead, unless death takes place from disease, and taken before any decomposition sets in, has chemically the same characteristics as that derived from animals slaughtered. If, however, the animals have been dead some time before rendering a considerable decomposition of the glycerides takes place and the amount of free acid in the fat is thus largely increased. Such fat also shows a distinctly unpleasant odor, by which it can readily be detected from genuine lard. Peanut oil and some other vegetable oils have also been mentioned as adulterants of lard. While it may be true that many attempts have been made to use the above substances in the adulteration of lard on a small scale, it is also quite

true that such attempts have never attained any importance from a commercial point of view.

(6) PROPERTIES OF ADULTERATED LARDS.

In external appearances to an unskilled person adulterated lards are not appreciably different from the pure article. An expert, however, is generally able to tell, by taste, odor, touch, and grain, a mixed lard from a pure one. There is usually enough lard in the adulterated article to give to it the taste and odor of a genuine one. Mixtures of fat, however, have been made, and perhaps sold as lard, which contained no hog grease whatever.* In the following descriptions an endeavor has been made to give the chief characteristics of an adulterated lard on the same plan as the descriptions of pure lard and the adulterations thereof which precede.

A.—PHYSICAL PROPERTIES.

(a) *Specific gravity*.—But little stress can be laid upon the numbers representing the specific gravity of adulterated lards since the materials of which they are composed have nearly the same specific gravity as the pure article. The addition of cotton oil, however, raises the specific gravity, and when this substance is present in quantities above 15 per cent. its influence on the specific gravity of the sample is marked. At 35° the specific gravity of adulterated lards varies from .906 to .910, compared with water at same temperature.

(b) *Melting point*.—The melting point of the adulterated lards is in most cases nearly the same as that of pure lards, but in some samples lower. This arises from the fact, which has already been noticed, of the low melting point of the cotton oil, which is one of the principal adulterants used. The numbers representing the melting points of adulterated lards, which will be found in the following tables, emphasize the fact which has already been noted that the lowering of the melting point is not theoretically proportional to the content of cotton oil found in the adulterated lards of commerce. In a number of samples of lards containing cotton oil from Fairbank & Co. the lowest melting point found was 31.3°, and the highest 41.9°, and the mean 38.1°. In the series of samples from Armour & Co. the lowest melting point noticed was 38.9°, and the highest 43.3°, and the mean 40.6°. The melting point of the Armour samples approaches much nearer that of pure kettle rendered lard than those received from Fairbank & Co. the latter being nearly the same as for steam lards. Although the melting point is not of itself a property of very great importance from an analytical point of view, yet its determination should never be neglected in a comprehensive analytical examination.

(c) *Color reaction*.—The amount of coloration shown by an adulterated

* *Cotolene* is a mixture of cotton oil and oleo-stearine, prepared by N. K. Fairbank & Co. It is sold under its true name and not as lard.

lard when treated with sulphuric or nitric acid, depends chiefly upon the percentage of cotton oil which it contains. Since from a commercial point of view the introduction of a small amount of cotton oil would not prove profitable, we find in the adulterated lards of commerce, as a general rule, strong color reactions. It might be possible, however, to mix with a pure lard so small a quantity of cotton oil as to render doubtful to the analyst the character of the color reaction produced. Some of the colors produced in the adulterated lards examined, as copied from the note-books, are as follows: "light brown," "pink red brown," "light yellow red," "light pink," "deep brown," "red," "deep red brown," etc. The appearance of a pinkish tint is often found in adulterated lards containing a notable portion of beef-fat stearine, although this coloration is not considered a certain indication of the presence of this substance.

(d) *Refractive index*.—The refractive index of the mixed lards naturally varies with the proportion of cotton oil which may be present. The greater the quantity of cotton oil the higher the refractive index. The refractive index of the Armour mixed lards is decidedly lower than that of the Fairbank samples. The following is the number representing the mean refractive index of the Armour samples at 25° , viz, 1.4634. The number representing the mean refractive index of the Fairbank samples is 1.4651. The refractive index is a much more important property in the sorting of lards than the melting point.

(e) *Rise of temperature with sulphuric acid*.—As is to be expected, we find here also great variation, depending on the nature and the quantity of the adulterants present. The presence of tallow stearine tends to diminish the rise of temperature with sulphuric acid, while cotton-oil has the opposite effect. As the relative proportion of these two ingredients and also the amount of pure lard varies, we may expect corresponding variation in the temperature shown on mixing the lard with sulphuric acid. In the samples of Armour's lards examined, the highest rise of temperature noticed was 58.9° and the lowest 42.1° . This latter number is almost identical with that furnished with pure lards. In Fairbank's lards the least rise of temperature noticed was 51.3° and the greatest 68.8° . These numbers show a larger proportion of cotton oil in the Fairbank than in the Armour samples. This rise of temperature as a diagnostic sign is valuable, and its determination should never be omitted.

(f) *Crystallization point of fatty acids*.—In Armour's lards the mean temperature of crystallization for the fat acids was found to be 39.8° . In the Fairbank lards it was 37.4° .

(g) *Melting point of fat acids*.—The mean melting point of the fat acids in the Armour samples was 42.8° . In the Fairbank samples it was 40.6° .

B.—CHEMICAL PROPERTIES.

(a) *Volatile acids*.—The remark which has been made in regard to the volatile acids of pure lards and their adulterants is also applicable for mixed lards. The amount is so minute as to be of no value from an analytical point of view.

(b) *Saponification equivalent*.—The numbers representing the saponification equivalent do not afford any particular indication of the kind of adulteration used. In the samples of Fairbank mixed lards examined the mean saponification equivalent found was 279.4. In the Armour samples it was 275.

(c) *Iodine number*.—The amount of iodine absorbed by a mixed lard gives a valuable indication of the kind of the ingredients which have been added to it. It has already been seen that the stearines, especially those derived from tallow, have a very low iodine number, while cotton-seed oil has a very high one. It is therefore possible to mix these two substances together so that the resulting iodine number may be about the same as that of pure lard, viz, 60 per cent. In the samples of the Armour mixed lards examined the mixture seems to have been made in about the proportion indicated. The lowest iodine number observed in these lards was 54.11 per cent., which is decidedly less than that of normal pure lard. The highest number observed was 71.19 per cent. The other numbers were slightly above those obtained for pure lard. In the samples of mixed lards from Fairbank & Co. the iodine numbers are much higher. The lowest number observed was 78.24 and the highest 94.78 per cent.

(d) *Reaction with nitrate of silver*.^{*}—Mixed lards containing cotton oil show a reduction of metallic silver in a greater or less degree, according to the proportion of cotton oil present. In every case where cotton oil was known to be present in a mixed lard this reaction was noticed. It would be possible, however, to put so small a portion of cotton oil into a lard as to render difficult the positive detection of it by the nitrate of silver test.

(e) *Microscopic appearances*.—The mixed lards, under the conditions described further on, show in the field of vision of the microscope distinct tufted crystals of the stearines which have been used as adulterants. The rhombic crystals of pure lard are also often noticed in this field.

(f) *Moisture in mixed lards*.—Mixed lards generally contain only a trace of water. In one instance, however, water appears to have been added as an adulterant, over 30 per cent. of it having been found. The use of water as an adulterant of lard, however, is not common.

^{*} Later observations show that in samples kept for several months the reaction with nitrate of silver is indistinct and in some cases entirely absent.

Comparison of properties of lard and compound lards.

The mean results of the analytical data are as follows:

Kind of samples.	Specific gravity.	Saponification equivalent.	Melting point of the glycerides.	Melting point of the fat acids.	Crystallizing point of fat acids.	Rise of temperature.	Percentage of iodine absorbed.	Refractive index.
Pure lard9053	280.3	○	○	○	○	62.48	1.4620
Lard of miscellaneous origin9067	274.4	41.7	42.9	39.6	45.7	64.34	1.4633
Prime steam lard9055	283.5	37.0	42.1	38.6	39.9	62.66	1.4623
Armour's lards9060	275.0	40.6	42.8	39.8	46.5	63.58	1.4624
Fairbank's lards9095	279.4	38.1	40.6	37.4	57.9	85.31	1.4651

STATISTICS OF THE LARD INDUSTRY.

It was developed in the investigations before the Committees on Agriculture of the Senate and House of Representatives that the annual production of lard in the United States is 600,000,000 pounds, of which about half is pure lard and the other half pure lard mixed with stearine and cotton oil, the "refined" or compound lard of commerce. The annual exports of lard are about 320,000,000 pounds, of which about 40 per cent. were compound or refined lard.*

According to the figures furnished by the Bureau of Statistics, the production of lard from 1877 to 1887, inclusive, was as follows:

Years.	Total.	Years.	Total.
	<i>Pounds.</i>		<i>Pounds.</i>
1886-'87	527,032,000	1881-'82	468,929,000
1885-'86	514,230,000	1880-'81	517,660,000
1884-'85	480,405,000	1879-'80	479,020,000
1883-'84	444,450,000	1878-'79	514,295,000
1882-'83	419,513,000	1877-'78	404,572,000

* Statement of Mr. G. H. Webster before House Committee on Agriculture. Report of Hearings, p. 26.

The exports from 1873 to 1888 are shown by the following numbers :

Years.	Lard exported.	Years.	Lard exported.
	<i>Pounds.</i>		<i>Pounds.</i>
1873.....	234, 901, 511	1881.....	335, 001, 686
1874.....	184, 100, 226	1882.....	239, 904, 657
1875.....	167, 579, 377	1883.....	273, 236, 610
1876.....	198, 008, 212	1884.....	228, 165, 733
1877.....	237, 744, 307	1885.....	301, 305, 105
1878.....	345, 603, 527	1886.....	298, 083, 094
1879.....	343, 119, 208	1887.....	324, 515, 224
1880.....	405, 436, 658		

If we take the percentage of cotton oil in the compound lard at 40, the total weight of oil used in manufacturing mixed lard is 120,000,000 pounds.

In addition to this, large quantities of cotton oil are used for salad dressing and culinary operations and in the manufacture of a substitute for lard, cotelene, which contains no hog grease whatever.

METHODS OF ANALYSIS EMPLOYED.

The processes employed in conducting the analytical work, the results of which follow, will now be briefly described.

METHOD OF TAKING THE SPECIFIC GRAVITY.

(a) *By the pycnometer.*—Two kinds of specific gravity flasks have been used in the determinations of the specific gravities, as represented in Fig. 17, viz, a plain flask with a stopper having a capillary perforation and a flask carrying a stopper to which is attached a delicate thermometer. If the specific gravity is to be taken at a temperature of 100° or that of boiling water, the plain flask is preferable; if, however, it is to be taken at some temperature below that point, for instance, 40°, the flask with the thermometer is used. The manipulation in both cases is the same.

(b) *Graduation of the flasks.*—The flasks, having been cleaned, are rinsed with alcohol and ether and thoroughly dried, care being taken that the ether and alcohol vapors are removed from the interior of the flask. The flask, after it is cleaned, should be handled with dry fingers or with forceps. The stopper having been inserted, the dried and cleaned flask is weighed empty at the temperature of the balance room. If the flask be wiped with a silk handkerchief or towel before weighing it should be allowed to stand fifteen minutes in the balance before the final weights are taken. The flask is now filled with recently boiled distilled water which, to avoid mixing with air, has not been shaken. It is placed in a bath of distilled water in a vessel with a flat bottom. The bath should contain as much water as is possible to avoid flowing

into the open neck of the flask. If the bath is to be kept at the boiling temperature the flask should be held steady by a wire attached to the edges of the vessel or by some other means. If the specific gravity is to be taken at a lower temperature than boiling water, say 40° , the flask having been filled with distilled water at a temperature below 40° , as described above, is closed with the stopper carrying the thermometer, which is pressed firmly to its place, care being taken that no air bubbles are occluded. The temperature of the bath is then raised slowly until it reaches 40° to 41° . The temperature of the bath is taken with another thermometer. The thermometer of the flask is carefully watched, especially as it approaches the required point. When the

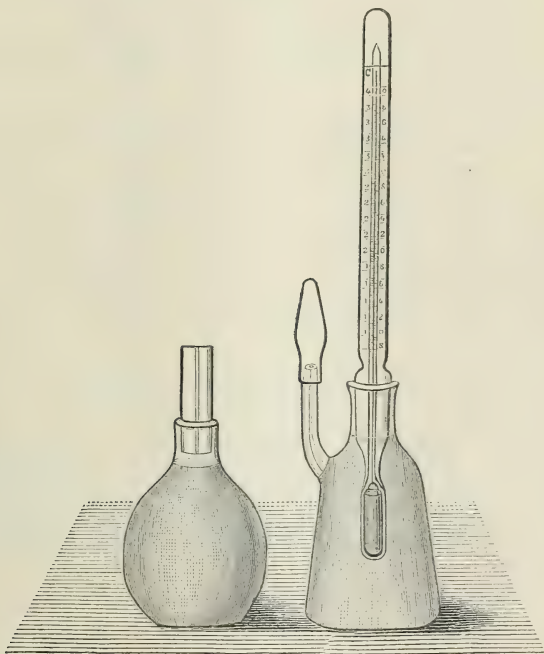


FIG. 17.

temperature of the bath is only slightly above that required the final temperature is reached only after some time, usually about one-half hour. The moment the required temperature is reached any water on top of the capillary tube is removed with blotting paper, the cap is placed upon the capillary tube and the pycnometer taken from the bath; it is at once wiped perfectly dry and placed in the balance, where it is allowed to remain until the temperature indicated by the thermometer is sensibly that of the balance room; it is then weighed and the weight of distilled water which it contains at that temperature determined. When the determination is to be made at the temperature of boiling water the specific-gravity flask is secured in the bath as indicated and

filled with recently boiled distilled water. The water in the bath is then brought to the boiling point by means of a lamp and the boiling continued for one half hour. Any evaporation which may take place from the specific-gravity flask is replaced by adding a few drops of boiling distilled water. At the end of the half hour the stopper of the flask is quickly inserted and firmly pressed into its position, any water remaining on the top of the stopper being removed by a piece of filter paper. The flask is then removed from the bath, wiped perfectly dry, placed in the balance and weighed as soon as it reaches the temperature of the balance room. The weight of the distilled water which the flask contains at the given temperature having been determined, the flask is rinsed with alcohol and ether and dried as in the first instance. It is then filled with the fat, the specific gravity of which is to be determined, with the same precautions as were used in determining the weight of water.*

Example of specific gravity of fat, at 100° (boiling distilled water).

	Grams.
Weight of flask, empty.....	11.0956
Weight of flask+water at 100°	39.6216
Weight of water.....	28.5260
Weight of flask with fat at 100°	36.8691
Weight of fat.....	25.7635
Specific gravity= $25.7635 \div 28.5260 = .90316$.	

(c) *By the Westphal balance.*—The specific gravity of a fat can be accurately determined by a modification of the balance known as the Westphal. This instrument is shown in Fig. 18.

The principle of the apparatus may be briefly stated as follows: A glass bob is so adjusted as to be capable of displacing a given number of grames, five, for instance, of distilled water at a given temperature when wholly immersed in the liquid and suspended by a fine platinum wire. These bobs may be had graduated for any temperature, but most conveniently for those already named, viz, 35° or 40° and 100°. It is necessary for accurate work with this instrument that the temperature of the fat or oil, the specific gravity of which is to be determined, should be exactly that for which the bob is graduated, as even a slight variation from the prescribed temperature will produce a serious error in the result. In order to secure greater accuracy, especially for taking specific gravities at a temperature of 40°, a fine analytical balance can be substituted for the Westphal instrument. Such a balance arranged for use in this way is represented in Fig. 19. It is inconvenient, however,

*To facilitate the escape of any occluded air in placing the stoppers in the flasks, I have had the stoppers constructed with a concave bottom, the center of the concavity being at the opening of the capillary tube. The top of the stopper is also ground to a fine edge, so that any liquid that may issue from the capillary tube may flow away and thus escape absorption,

to use the ordinary balance for this method for temperatures near the boiling point on account of the difficulty of conducting the condensed vapors out of the balance case. For our work, therefore, we have used this balance only for lower temperatures.

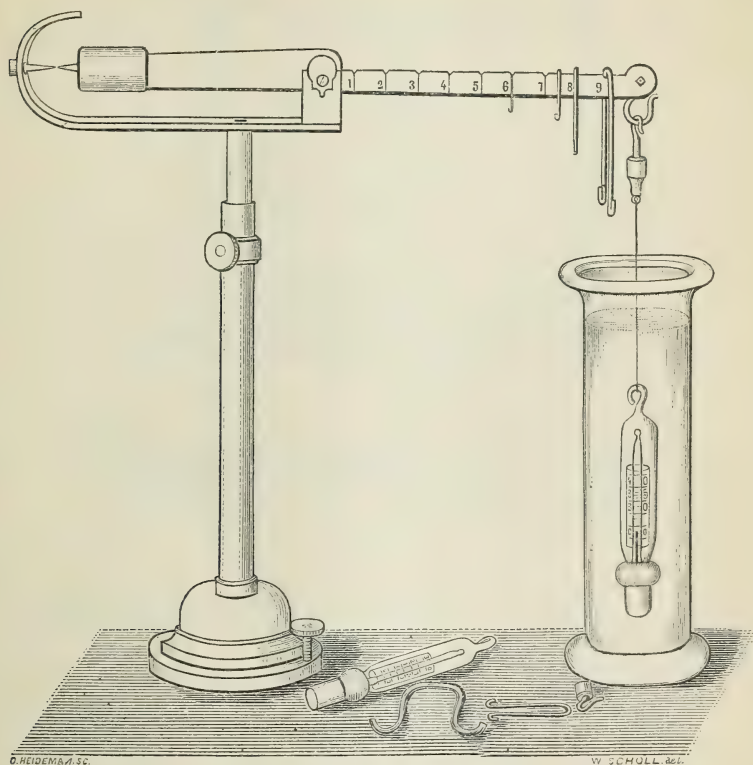


FIG. 18.

DESCRIPTION OF INSTRUMENT.

The Mohr or Westphal balance is well illustrated in the figure. The position of the instrument is shown in equilibrium. The bob is furnished with a delicate thermometer. If the bob be graduated for the displacement of exactly 5 grams of distilled water at 35° , for instance, a deep red line indicates that point. The weights are determined on the principle of the ordinary rider. There is one weight for the 5 grams and one for each 5 of decimal places of the under gram weight. The beam is so adjusted as to be in exact equilibrium when the dry bob is suspended in air. It is divided into ten parts. The big weight counts 5 when placed directly over the suspension point of the bob; 4.5 when placed at 9; 4.0 when placed at 8, etc.; when a lighter weight falls

on the same figure with a heavier it is suspended from the hook of the latter.

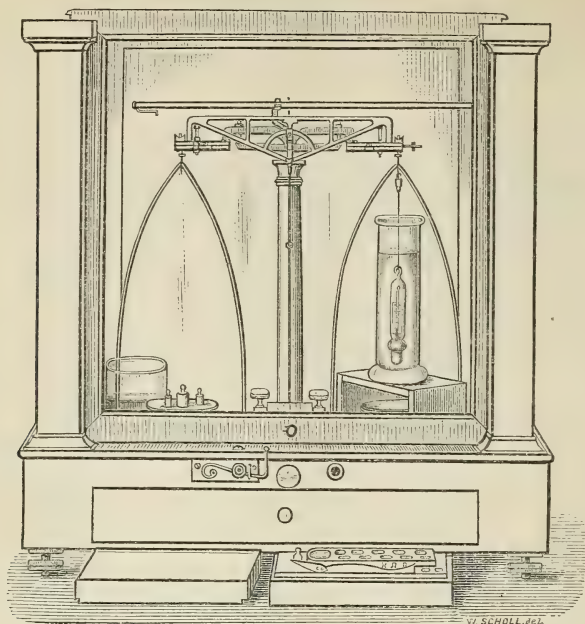


FIG. 19.

For liquids lighter than distilled water the numbers on the beam may be taken to represent the specific gravity.

Example.

Let the 5 g weight be at 9.
 the .5 g weight be at 1.
 the .05 g weight be at 4.
 the .005 g weight be at 5.

If the beam is in equilibrium at this disposition of the weights and the temperature of the liquid that of the red mark on the bob, the specific gravity would be .9145. The actual weight of liquid displaced would be 4.5725 g, which divided by 5 = .9145.

Before beginning work with the balance the bob should be carefully graduated in pure distilled water, recently boiled and at the required temperature. Any variation in the caliber of the bob is thus determined, and any necessary correction can be introduced into the result obtained.

To change the expression of the specific gravity from direct comparison with water at any given temperature to the standard of water at 15.5° or 4°, the factor of the co-efficient of expansion of water must be introduced. One cubic centimeter of water at 35° weighs 99.418 g.

Therefore a bob which displaces 5 g of water at 35° has a volume 5.029cc. This volume of water at 4° would weigh, therefore, 5.029 g. The above specific gravity referred to water at 4° would be $4.5725 \div 5.029 = .9092$.

In tabular form the above data are as follows:

	Grams.
Weight of 5.029cc oil at 35°	4.5725
Weight of 5.029cc water at 35°	5.0000
Weight of 5.029cc of water at 4°	5.0290
Relative weight of oil at 35° to water at 35° equals9145
Relative weight of oil at 35° to water at 4° equals9092

The change in volume of a fat or oil for each degree of temperature is approximately .0007cc for each cubic centimeter of the oil. The weight of a given volume of an oil having been determined at any temperature, its weight at the required temperature can be approximately calculated.

Thus, in the above case—

	Grams.
5.029cc of oil at 35° weighs	4.5725
Then 5.029cc of oil at 4° weighs	4.6707
Then relative weight of oil at 4° to water at 4° equals9307
Then relative weight of oil at 4° to water at 35° equals9342

ESTIMATION OF THE SPECIFIC GRAVITY OF FATS, STEARINES, ETC., IN A SOLID CONDITION.*

(a) *Estimation of specific gravity at zero.*—A platinum crucible containing about 20cc is furnished with a fine platinum bail, which is fastened through two small holes drilled into the crucible at opposite points near the upper edge. To the handle of the crucible at the central point is fastened a fine platinum wire, furnished with a loop above, by means of which it is suspended from the hook of the balance. The crucible is weighed empty and then in water at zero. This is accomplished in the following way:

The pan of the balance is protected by a wooden bench in the ordinary way in taking specific gravities, and on this bench is placed a large beaker glass containing a smaller one. The space between the two beakers is filled with finely-powdered ice and the small beaker is nearly filled with distilled ice-water. The platinum crucible is suspended at such a height as to allow it to be wholly immersed in the water, including the bail and a small portion of the suspending platinum wire. The weight of the crucible having been determined in the water, it is taken out, carefully dried, and about 15 grams of the filtered and melted fat placed in it. The fat is allowed to solidify slowly at ordinary temperatures. The crucible with fat is then weighed in the air and placed in

* Wollny, Milch Zeitung, 1888, No. 25 et seq.

the ice-cold water as before and weighed. Before weighing it should be allowed to stand for one hour in the water at zero.

Let t' represent the weight of the empty crucible in the air.

Let t'' represent the weight of the empty crucible in the water.

Let b' represent the weight of the filled crucible in the air.

Let b'' represent the weight of the filled crucible in the water.

Let S represent the specific gravity. Then S is computed as follows :

$$S = \frac{b' - t'}{b' - b'' - t' + t''}.$$

(b) In the same manner the specific gravity can be computed at 15°, 20° or 25°, or at any higher temperature at which the fat or stearine will remain in solid condition.

(c) *Specific gravity by Sprengel's tube.*—(For account of this method of procedure consult Allen's Commercial Organic Analysis, vol 1, page 5.)

Much confusion has arisen concerning the real meaning of the specific gravities reported for lards and lard adulterants because of failure on the part of the authors to state all the conditions. All statements of specific gravities should be accompanied by the temperature at which they were taken and the temperature of the equal volume of water with which they are compared. It would be convenient if some uniform practice of stating specific gravities could be adopted by all analysts.

(d) *Notes on methods of computing specific gravities.*—The rates of expansion of lard and the fat oils used as lard were carefully studied by Dr. C. A. Crampton, and I insert here his observations thereon.

All the determinations were made very carefully by the methods described, and the figures given are in all cases the average of two or more duplicates. In the densities taken at low temperatures the flasks filled with the samples were placed in a vessel containing water somewhat above the temperature at which the determination was to be made, and when it had dropped to this point they were carefully stoppered, taken out of the vessel, allowed to cool, and weighed. The determinations at high temperature were made by placing the flasks in an oil bath. The heat was raised as high a point as was deemed safe, and at the temperatures used, 190° to 200° C., there was scarcely a darkening of the contents of the flasks, and I am convinced that no decomposition had taken place which would alter appreciably the density of the sample. From the densities taken at these two widely different temperatures the mean increase in density and the mean co-efficient of expansion was determined for each sample.

The formula used for this was the one usually given in the books :*

$$\delta = \frac{D_o - D_o'}{(t' - t)D_o'}$$

in which

D_o = density at the lower observed temperature.

D_o' = density at the higher observed temperature.

t = lower temperature.

t' = higher temperature.

* Watts Dictionary, Vol. III, p. 71.)

Although it appears to me that the formula as follows would be more correct—

$$\frac{D_0 - D_0'}{(t - t') D_0}$$

or, still better:

$$\frac{D_0 - D_0'}{(t' - t) \times \frac{D_0 \times D_0'}{2}}$$

Great confusion exists in chemical literature in the expression of specific gravities. I have referred all my results to water at 4° C., believing that eventually all specific gravities will be stated in these terms, as is the custom in continental Europe.

The absolute densities are calculated from the formula $\Delta = \delta + \kappa$, in which

Δ = Co-efficient of absolute expansion.

δ = Co-efficient of apparent expansion in glass.

κ = Co-efficient of cubical expansion of glass = .000025.

The weights were not reduced to a vacuum, and no correction was made for the thread of mercury projecting above the bulb. The specific gravities of the different samples at various temperatures are also given in the last columns of the table, these having been calculated by means of the co-efficients of expansion.

Specific gravities of fats and oils at various temper-

Serial number.	Description.	Temperature.	Apparent specific gravity in glass vessels.	Absolute specific gravity.	Temperature.	Apparent specific gravity in glass vessels.
<i>Lards.</i>						
5674	Leaf lard rendered in laboratory U. S. Department Agriculture	$\begin{smallmatrix} 0^{\circ} C. \\ \{ +40 \} \\ \{ +4 \} \end{smallmatrix}$.89679	.89709	$\begin{smallmatrix} 0^{\circ} C. \\ \{ +190 \} \\ \{ +4 \} \end{smallmatrix}$.80475
5673	Intestinal lard rendered in laboratory U. S. Department Agriculture	$\begin{smallmatrix} \{ +40 \} \\ \{ +4 \} \end{smallmatrix}$.89635	.89725	$\begin{smallmatrix} \{ +190 \} \\ \{ +4 \} \end{smallmatrix}$.80315
5672	Head lard rendered in laboratory U. S. Department Agriculture	$\begin{smallmatrix} \{ +40 \} \\ \{ +4 \} \end{smallmatrix}$.89816	.89906	$\begin{smallmatrix} \{ +190 \} \\ \{ +4 \} \end{smallmatrix}$.80512
5591	Squires' pure lard, made by J. P. Squires & Co., Boston, Mass	$\begin{smallmatrix} \{ +40 \} \\ \{ +4 \} \end{smallmatrix}$.89700	.89790	$\begin{smallmatrix} \{ +95 \} \\ \{ +4 \} \end{smallmatrix}$.80319
5606	Cassard's pure lard, made by Cassard & Co., Baltimore, Md	$\begin{smallmatrix} \{ +40 \} \\ \{ +4 \} \end{smallmatrix}$.89848	.89938	$\begin{smallmatrix} \{ +187 \} \\ \{ +4 \} \end{smallmatrix}$.80888
5610	Armour's compound lard, made by Armour & Co., Chicago, Ill.	$\begin{smallmatrix} \{ +40 \} \\ \{ +4 \} \end{smallmatrix}$.89940	.90030	$\begin{smallmatrix} \{ +190 \} \\ \{ +4 \} \end{smallmatrix}$.80946
5611	Armour's compound lard, made by Armour & Co., Chicago, Ill.	$\begin{smallmatrix} \{ +40 \} \\ \{ +4 \} \end{smallmatrix}$.89854	.89944	$\begin{smallmatrix} \{ +190 \} \\ \{ +4 \} \end{smallmatrix}$.80522
5646	Fairbank's compound lard, made by Fairbank & Co., Chicago, Ill.	$\begin{smallmatrix} \{ +40 \} \\ \{ +4 \} \end{smallmatrix}$.90000	.90090	$\begin{smallmatrix} \{ +190 \} \\ \{ +4 \} \end{smallmatrix}$.80724
<i>Lard stearines.</i>						
5613	Lard stearine used in Armour's compound lard	$\begin{smallmatrix} \{ +50 \} \\ \{ +4 \} \end{smallmatrix}$.88836	.88951	$\begin{smallmatrix} \{ +200 \} \\ \{ +4 \} \end{smallmatrix}$.79579
5643	Lard stearine used in Fairbank's compound lard	$\begin{smallmatrix} \{ +50 \} \\ \{ +4 \} \end{smallmatrix}$.88850	.88965	$\begin{smallmatrix} \{ +200 \} \\ \{ +4 \} \end{smallmatrix}$.79677
<i>Beef fat and oleo-stearines.</i>						
5607	Pure beef fat from the testicle, obtained from Prof. S. P. Sharpless	$\begin{smallmatrix} \{ +50 \} \\ \{ +4 \} \end{smallmatrix}$.88998	.89113	$\begin{smallmatrix} \{ +190 \} \\ \{ +4 \} \end{smallmatrix}$.80289
5612	Oleo-stearine, used in Armour's compound lard	$\begin{smallmatrix} \{ +50 \} \\ \{ +4 \} \end{smallmatrix}$.88615	.88730	$\begin{smallmatrix} \{ +200 \} \\ \{ +4 \} \end{smallmatrix}$.79487
5644	Oleo-stearine, used in Fairbank's compound lard	$\begin{smallmatrix} \{ +50 \} \\ \{ +4 \} \end{smallmatrix}$.88523	.88638	$\begin{smallmatrix} \{ +200 \} \\ \{ +4 \} \end{smallmatrix}$.79286
<i>Cottonseed stearine.</i>						
5675	Cottonseed stearine, obtained from Prof. D. Wesson ..	$\begin{smallmatrix} \{ +40 \} \\ \{ +4 \} \end{smallmatrix}$.90313	.90403	$\begin{smallmatrix} \{ +190 \} \\ \{ +4 \} \end{smallmatrix}$.80689
<i>Cottonseed oils.</i>						
5687	Crude cottonseed oil, obtained from Prof. D. Wesson ..	$\begin{smallmatrix} \{ +23 \} \\ \{ +4 \} \end{smallmatrix}$.91548	.91595	$\begin{smallmatrix} \{ +190 \} \\ \{ +4 \} \end{smallmatrix}$.81118
5682	Crude cottonseed oil, obtained from Southern Cotton Oil Trust	$\begin{smallmatrix} \{ +23 \} \\ \{ +4 \} \end{smallmatrix}$.91744	.91791	$\begin{smallmatrix} \{ +190 \} \\ \{ +4 \} \end{smallmatrix}$.81573
5683	Summer yellow cottonseed oil, obtained from Southern Cotton Oil Trust	$\begin{smallmatrix} \{ +23 \} \\ \{ +4 \} \end{smallmatrix}$.91589	.91636	$\begin{smallmatrix} \{ +190 \} \\ \{ +4 \} \end{smallmatrix}$.81021
5684	Summer white cottonseed oil, obtained from Southern Cotton Oil Trust	$\begin{smallmatrix} \{ +23 \} \\ \{ +4 \} \end{smallmatrix}$.91578	.91625	$\begin{smallmatrix} \{ +190 \} \\ \{ +4 \} \end{smallmatrix}$.80959
5685	Winter yellow cottonseed oil, obtained from Southern Cotton Oil Trust	$\begin{smallmatrix} \{ +24 \} \\ \{ +4 \} \end{smallmatrix}$.91623	.91673	$\begin{smallmatrix} \{ +190 \} \\ \{ +4 \} \end{smallmatrix}$.80525
5686	Winter white cottonseed oil, obtained from Southern Cotton Oil Trust	$\begin{smallmatrix} \{ +24 \} \\ \{ +4 \} \end{smallmatrix}$.91636	.91686	$\begin{smallmatrix} \{ +190 \} \\ \{ +4 \} \end{smallmatrix}$.81017
5645	Refined cottonseed oil used in Armour's compound lard	$\begin{smallmatrix} \{ +23 \} \\ \{ +4 \} \end{smallmatrix}$.91667	.91714	$\begin{smallmatrix} \{ +190 \} \\ \{ +4 \} \end{smallmatrix}$.80925
<i>Olive oils.</i>						
5617	Pure olive oil, obtained from Z. D. Gilman, Washington, D. C.	$\begin{smallmatrix} \{ +23 \} \\ \{ +4 \} \end{smallmatrix}$.91079	.91126	$\begin{smallmatrix} \{ +190 \} \\ \{ +4 \} \end{smallmatrix}$.80430
5624	Pure olive oil, obtained from Prof. S. P. Sharpless ..	$\begin{smallmatrix} \{ +23 \} \\ \{ +4 \} \end{smallmatrix}$.91034	.91081	$\begin{smallmatrix} \{ +190 \} \\ \{ +4 \} \end{smallmatrix}$.80538

atures, with mean co-efficient of expansion.

Absolute specific gravity.	Range in temperature.	Mean difference in specific gravity for each degree centigrade (apparent in glass vessels).	Mean co-efficient of expansion (apparent in glass vessels).	Mean co-efficient of expansion (absolute).	Specific gravity (apparent).				Serial number.
					$d = \frac{+15.50}{+40}$	$d = \frac{+400}{+40}$	$d = \frac{+500}{+40}$	$d = \frac{+1000}{+40}$	
	° C.								
.80940	{ + 40 } { + 190 }	.0006136	.0007624	.0007874	.91181	.89679	.89065	.85997	5674
.80789	{ + 40 } { + 190 }	.0006213	.0007726	.0007986	.91157	.89635	.89014	.85997	5673
.80977	{ + 40 } { + 190 }	.0006203	.0007704	.0007954	.91336	.89816	.89195	.86094	5672
.89546	{ + 40 } { + 95 }	.0006147	.0007122	.0007372	.91206	.89700	.89085	.86012	5591
.81345	{ + 40 } { + 187 }	.0006095	.0007535	.0007785	.91341	.89848	.89238	.86191	5606
.81111	{ + 40 } { + 190 }	.0006196	.0007683	.0007933	.91458	.89940	.89320	.86222	5610
.80987	{ + 40 } { + 150 }	.0006221	.0007726	.0007976	.91378	.89854	.89232	.86121	5611
.81189	{ + 40 } { + 190 }	.0006184	.0007660	.0007910	.91515	.90000	.89382	.86289	5646
.80069	{ + 50 } { + 200 }	.0006171	.0007755	.0008005	.90965	.89453	.88836	.85750	5613
.80167	{ + 50 } { + 200 }	.0006115	.0007675	.0007925	.90959	.89461	.88850	.85792	5643
.80754	{ + 50 } { + 190 }	.0006221	.0007748	.0007998	.91144	.89620	.88998	.85888	5607
.79977	{ + 50 } { + 200 }	.0006085	.0007656	.0007906	.90714	.89223	.88615	.85572	5612
.79776	{ + 50 } { + 200 }	.0006158	.0007767	.0008017	.90647	.89138	.88523	.85444	5644
.81154	{ + 40 } { + 190 }	.0006416	.0007951	.0008201	.91884	.90313	.89671	.86463	5675
.81583	{ + 23 } { + 190 }	.0006245	.0007699	.0007949	.92016	.90486	.89862	.86739	5687
.82038	{ + 23 } { + 190 }	.0006080	.0007466	.0007716	.92200	.90708	.90090	.87054	5682
.81486	{ + 23 } { + 190 }	.0006328	.0007813	.0008063	.92063	.90514	.89880	.86716	5683
.81424	{ + 23 } { + 190 }	.0006359	.0007858	.0008108	.92055	.90497	.89861	.86681	5684
.80990	{ + 24 } { + 190 }	.0006685	.0008302	.0008552	.92191	.90553	.89885	.86542	5685
.81482	{ + 24 } { + 190 }	.0006397	.0007896	.0008146	.92179	.90612	.89972	.86774	5686
.81390	{ + 23 } { + 190 }	.0006462	.0007943	.0008193	.92150	.90573	.89930	.86714	5645
.80885	{ + 23 } { + 190 }	.0006377	.0007928	.0008178	.91557	.89995	.89351	.86168	5617
.81003	{ + 23 } { + 190 }	.0006285	.0007801	.0008054	.91505	.89965	.89337	.86194	5624

It will be seen that the results confirm, in the main, Allen's* conclusions in regard to the generally uniform rate of expansion of all fats and oils. The average increase in density in my samples would be rather lower than the figure he gives (.00064) for each degree C., as his figure was evidently calculated from the formula

$$\delta = \frac{D_0 - D_0'}{(t' - t) D_0}$$

Kopp† gives a figure very nearly the same as mine, for the mean absolute expansion co-efficient of olive oil, viz: .00080348.

In the lards and oils in the above table, I also determined the density by the plummet at +35° C. These results, together with results calculated from the flask determinations, so as to make the figures comparable, are given in the following table:

Comparison of results with plummet and with specific-gravity flask.

	With plummet, $d = +35^{\circ}\text{C.}$	With specific gravity flask, $d = +35^{\circ}\text{C.}$
<i>Lards:</i>		
1.....	.9058	.90508
2.....	.9047	.90484
3.....	.9066	.90632
4.....	.9044	.90523
5.....	.9067	.90671
6.....	.9069	.90797
7.....	.9068	.90667
8.....	.9080	.90816
<i>Cotton-seed oil:</i>		
1.....	.9151	.91391
2.....	.9164	.91510
3.....	.9134	.91377
4.....	.9132	.91357
5.....	.9154	.91412
6.....	.9138	.91475
7.....	.9133	.91472
<i>Olive oil:</i>		
1.....	.9076	.90848
2.....	.9080	.90811
Mean9098	.90985

This short series of comparisons adds testimony to the accuracy of the Archimedean method for taking specific gravities, and it is certainly a most rapid and convenient means to this end. I went through nearly the entire series of samples used in our lard investigation, about 150 in all, in three days. I would call attention to a slight inaccuracy in Allen's description of the method. On page 14 (Vol. II), he says: "The plummet should have a displacement of exactly 5cc (in water). This should, of course, be 5 *grams*, otherwise he would be comparing *volume* with *weight*, as it is the *weight* of the sample displaced which is used as the numerator of the fraction of which the *weight* of water displaced is the denominator in the expression of its specific gravity. The error would not be so great where the volume and density of water were taken as being identical at 15.5° C., but still quite an appreciable error would be introduced when the determination on the sample was made at 35° to 50° C., as on a fat, for example. There is much need for more exact mathematics in calculating specific gravities, and more uniformity in methods of expressing them among chemists is greatly to be desired.

* Op. cit., p. 20.

† Lieb. Ann., 93, p.129.

(b) *Melting point*.—The term melting point applied to a glyceride does not indicate a physical state capable of being appreciated with definiteness. As usually employed it indicates the temperature at which the fat becomes transparent; but this temperature, as is well known, varies under certain conditions chiefly dependent upon the initial temperature of the body. A more definite point, and one usually capable of being ascertained, is that where a thin disk of the fat, when freed from the attraction of gravitation and left to its own molecular forces, assumes a sensibly spherical state. The melting point given in the following analytical tables, with the exceptions to be noted, has been determined by an apparatus based on the above principle. This apparatus is described in the *Journal of Analytical Chemistry*, volume 1, part 1, pages 39 *et seq.*

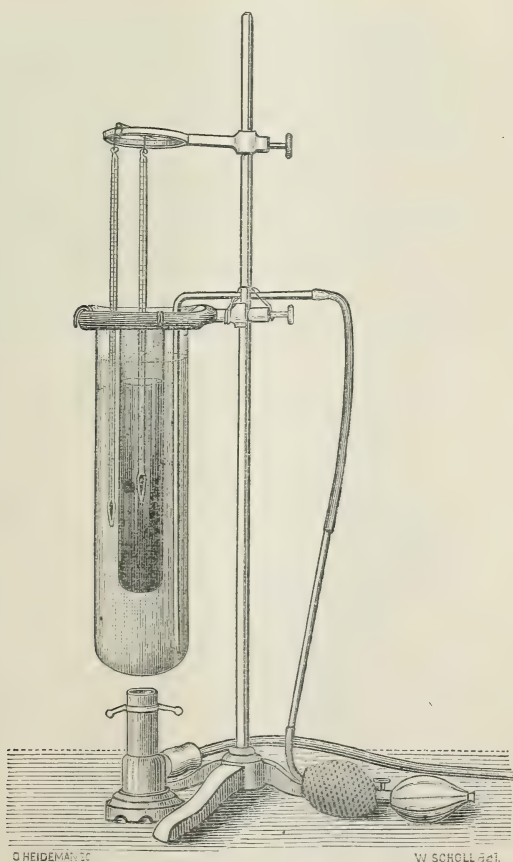


FIG. 20.

DESCRIPTION OF APPARATUS.

The apparatus, Fig. 20, consists of (1) an accurate thermometer for reading easily tenths of a degree; (2) a less accurate thermometer

for measuring the temperature of water in the large beaker glass; (3) a tall beaker glass, 35cm high and 10cm in diameter; (4) a test tube 30cm high and 3.5cm in diameter; (5) a stand for supporting the apparatus; (6) some method of stirring the water in the beaker, for example, a blowing bulb of rubber and a bent glass tube extending to near the bottom of the beaker; (7) a mixture of alcohol and water of the same specific gravity as the fat to be examined.

Manipulation.—The disks of the fat are prepared as follows: The melted and filtered fat is allowed to fall from a dropping tube from a height of 15 to 20 cm on to a smooth piece of ice floating in water. The disks thus formed are from 1 to $1\frac{1}{2}$ cm in diameter and weigh about 200 milligrams. By pressing the ice under the water the disks are made to float on the surface, whence they are easily removed with a steel spatula.

The mixture of alcohol and water is prepared by boiling distilled water and 95 per cent. alcohol for ten minutes to remove the gases which they may hold in solution. While still hot the water is poured into the test tube already described until it is nearly half full. The test tube is then nearly filled with the hot alcohol. It should be poured in gently down the side of the inclined tube to avoid too much mixing. If the tube is not filled until the water has cooled, the mixture will contain so many air bubbles as to be unfit for use. These bubbles will gather on the disk of fat as the temperature rises and finally force it to the top of the mixture.

The test tube containing the alcohol and water is placed in a vessel containing cold water, and the whole cooled to below 10° . The disk of fat is dropped into the tube from the spatula, and at once sinks until it reaches a part of the tube where the density of the alcohol—water is exactly equivalent to its own. Here it remains at rest and free from the action of any force save that inherent in its own molecules.

The delicate thermometer is placed in the test tube and lowered until the bulb is just above the disk. In order to secure an even temperature in all parts of the alcohol mixture in the vicinity of the disk the thermometer is moved from time to time in a circularly, pendulous manner. A tube prepared in this way will be suitable for use for several days; in fact, until the air bubbles begin to attach themselves to the disk of fat. In no case did the two liquids become so thoroughly mixed as to lose the property of holding the disk at a fixed point, even when they were kept for several weeks.

In practice, owing to the absorption of air, it has been found necessary to prepare new solutions every third or fourth day.

The disk having been placed in position, the water in the beaker glass is slowly heated and kept constantly stirred by means of the blowing apparatus already described.

When the temperature of the alcohol-water mixture rises to about 6° below the melting point the disk of fat begins to shrivel and gradually rolls up into an irregular mass.

The thermometer is now lowered until the fat particle is even with the center of the bulb. The bulb of the thermometer should be small, so as to indicate only the temperature of the mixture near the fat. A gentle rotary movement should be given to the thermometer bulb, which might be done with a kind of clock-work. The rise of temperature should be so regulated that the last 2° of increment require about ten minutes. The mass of fat gradually approaches the form of a sphere, and when it is sensibly so the reading of the thermometer is to be made. As soon as the temperature is taken the test tube is removed from the bath and placed again in the cooler. A second tube, containing alcohol and water, is at once placed in the bath. It is not necessary to cool the water in the bath. The test tube (ice-water being used as a cooler) is of low enough temperature to cool the bath sufficiently. After the first determination, which should be only a trial, the temperature of the bath should be so regulated as to reach a maximum about 1.5° above the melting point of the fat under examination.

Working thus with two tubes about three determinations can be made in an hour. After the test tube has been cooled the globule of fat is removed with a small spoon attached to a wire before another disk of fat is put in.

(d) *Refractive index*.—The apparatus used in determining the refractive index is one described by Professor Abbe in a brochure entitled “*Neue Apparate zur Bestimmung des Brechungs- und Zerstreuungsvermögens fester und flüssiger Körper*.” The apparatus is represented in Fig. 21. For lard it is necessary that the index of refraction be de-

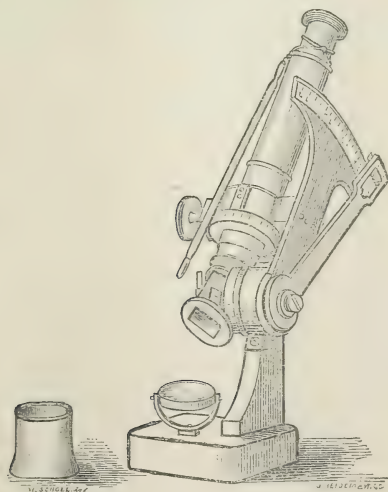


FIG. 21.

termined in a room where the temperature is higher than 30° and even higher than 35° . For determining the refractive index of oleo-stearines the temperature must be considerably above 40° . For very high tem-

peratures I used the hot-room of a Turkish bath establishment. In order that the fats might quickly come to the temperature of the room and also be in a convenient apparatus for dropping upon the paper holder they were kept in a U-shaped small tube-holder. The one arm of the tube was drawn out to an almost capillary diameter, bent over at the end forming a spout to facilitate the dropping of the oil upon the paper receptacle.

The apparatus is operated as follows: Fine tissue paper of rather heavy body is cut into rectangular pieces 3 cm in length by 1.5 cm in breadth. One of these pieces of paper is placed on the lower of the two glass prisms of the apparatus. Two or three drops of the oil or the fat are placed upon the paper and the upper prism carefully placed in position so as not to move the paper from its place. In charging the apparatus with the oil in this way it is placed in the horizontal position. After the paper disk holding the fat is secured by replacing the upper prism the apparatus is placed in its normal position and the index moved until the light directed through the apparatus by the mirror shows the field of vision divided into dark and light portions. The dispersion apparatus is now turned until the rainbow colors on the part between the dark and light field have disappeared. Before doing this, however, the telescope, the eye-piece of the apparatus, is so adjusted as to bring the cross-lines of the field of vision distinctly into focus. The index of the apparatus is now moved back and forth until the dark edge of the field of vision falls exactly in the intersection of the cross-lines. The refractive index of the fat under examination is then read directly upon the scale by means of a small magnifying glass. To check the accuracy of the first reading the dispersion apparatus should be turned through an angle of 180° until the colors have again disappeared and the scale of the instrument again read. These two readings should fall closely together, and their mean is the true reading of the fat under examination.

METHOD OF DETERMINING REFRACTIVE INDEX AT TEMPERATURES ABOVE THE NORMAL.

The refractive index of lards, stearines, etc., can not be taken at the ordinary room temperatures. The best method of securing the desired temperature is to place the instrument and samples in a room provided with suitable heaters for maintaining the temperature at a constant point, about 50° . Some stearines may require a slightly higher temperature. I have, in the absence of any such room in our laboratory, used the hot-rooms of the Turkish bath to good advantage. Another method suggested by Mr. Von Schweinitz has been employed. The instrument is placed on the top of an air-bath maintained at a constant temperature. The room must also be kept without change of temperature. The instrument should be allowed to remain on the bath for at least one hour before work is commenced. If necessary it can be protected with

a hood, the side next the window being provided with an opening for admitting the light, and the one next the operator being entirely open.

The thermometer should rest with its bulb as closely as possible applied to the metallic casing of the prisms of the instrument. The temperature marked by it is much lower than that of the space between the prisms occupied by the film of oil under examination. For purposes of comparative readings a cotton oil is used, the refractive index of which is carefully determined at 25°.

When the temperature of the refractometer on the air bath has become constant, the same cotton oil is placed on the prisms, and after waiting for 10 to 20 minutes for the same temperature to be established, the index at that temperature is read off. The lards, stearines, etc., are then examined at that temperature and reduced to the standard of 25° by the factor determined as above. After the introduction of each fresh sample the instrument is allowed to stand for 10 to 20 minutes in order to secure a uniform temperature for all the readings.

Example.

Cotton oil, No. 6258:		
R. I. at 25°	=	1.4674
R. I. at N°	=	1.4565
Factor,		.0109
External T°	=	49.5
Calculated internal T°	=	57.5

At these temperatures a large number of indices of lards, stearines, fat acids, etc., was taken.

No	Material.	Observed index.	Index corrected to 25°.
6263	Prime lard stearine.....	1.4505	1.4614
6262	Neutral lard	1.4505	1.4614
6264	Kettle-rendered lard	1.4500	1.4609
6260	Golden cotoleme	1.4540	1.4649
6257	Oleo oil	1.4495	1.4604
6256	Oleo-stearine.....	1.4475	1.4584
5626do.....	1.4470	1.4579
5681	White cotton-oil stearine	1.4550	1.4650
5680	Yellow cotton-oil stearine	1.4555	1.4664
5606	Pure lard fat acid.....	1.4445	1.4554
5566	Lard fat acid.....	1.4455	1.4564
5626	Oleo-stearine fat acid	1.4475	1.4585
5680	Cotton-oil stearine fat acid	1.4515	1.4624
5681	Cotton oil-stearine	1.4475	1.4587
5576	Compound lard fat acid.....	1.4455	1.4564

(c) *Rise of temperature.*—The rise of temperature which fats and oils undergo when mixed with sulphuric acid was determined in the following manner. The apparatus used is represented in Fig. 22. The idea of this piece of apparatus was derived from a description given by

Dr. W. Ramsay of an apparatus used by him in the determination of the molecular weights of nitrogen trioxide and nitric peroxide.* The rise of temperature is not wholly independent of the initial tempera-

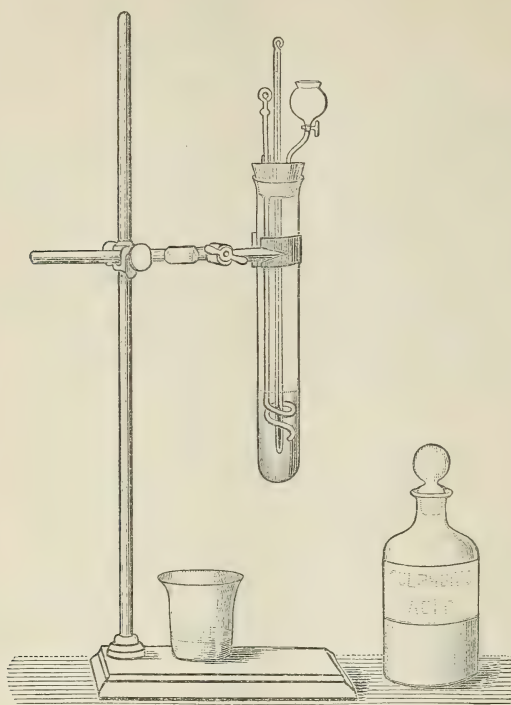


FIG. 22.

ture, and hence the initial temperature should be kept as nearly constant as possible; since most lards and adulterated lards melted at rather a high temperature are still liquid at 35° this temperature becomes a very convenient starting point. For oleo stearines the initial temperature should be 10° higher.

DESCRIPTION OF APPARATUS.

The test tube should be about 24 cm in length and 5 cm in diameter. It is furnished with a stopper with three holes, the one through the center carrying a delicate thermometer graduated to at least fifths of a degree. The second opening carries loosely a glass stirring-rod, which is bent into a coil at the lower extremity. This coil is so arranged as to have the thermometer pass through its center. The third perforation carries the funnel, which is bent outwards and upwards and holds the sulphuric acid.

* Journal of the Chemical Society, June, 1888, page 622. When in use the whole lower part of the apparatus is inclosed in a non-conducting case, as mentioned in the text.

Manipulation.—Fifty cc of the fat or oil to be examined are placed in the test tube and warmed or cooled, as the case may be, until the temperature is the one required for the beginning of the experiment, say 35°; 10 cc of the strongest sulphuric acid at the same temperature are placed in the funnel, the stopper being firmly fixed in its place; the test tube containing the oil is placed in a non-conducting receptacle; the wooden cylinder lined with cork, used in sending glass bottles by mail, I have found to be convenient for this purpose. A glass rod which fits loosely in the stopper, so as to be moved rapidly up and down, is held by the right hand of the operator; with his left hand he opens the glass stopcock of the funnel and allows the sulphuric acid to flow in upon the oil. The glass stirring-rod is now moved rapidly up and down for about 20 seconds, thus securing a thorough mixture of the oil and acid. The mercury rises rapidly in the thermometer and after two or three minutes reaches a maximum, and then, after two or three minutes more, begins to descend. The reading is made at the maximum point reached by the mercury. With pure cotton oil, linseed oil, and some other substances the rise of temperature is so great as to produce ebullition in the mass, causing it to foam up and fill the tube. To avoid this smaller quantities of acid should be used or the oil in question be diluted with a less thermogenic one, so that the maximum temperature may not be high enough to produce the effect noted.

I have thought that the value of this method of work might be increased by measuring the total temperature produced in mixing given quantities of fat and sulphuric acid, and hope soon to have a calorimeter constructed suitable for this purpose.

Experiments made with the apparatus described above in regard to the influence of the initial temperature have shown that a difference of 10° in the initial temperature would cause a difference of from 2° to 3° in the maximum temperature reached during the operation.

Prof. C. E. Munroe, of Newport, has made extensive experiments on the rise of temperature produced by the mixture of oils with sulphuric acid. He has published some of the results of his work in volume 10 of the Reports of the American Public Health Association.*

In a manuscript communication from the author under date of July 20, 1888, Professor Munroe makes the following additional observations upon his method and results:

I first sought speed in mixing, using a turn-table upon which the vessel was put, or mechanical stirrer placed in the vessel or shakers, etc., but while using Maumené's proportions there were discrepancies I could not explain. So then I varied the proportion until when I reached 20 cc of oil to 25 cc of H_2SO_4 (sp. pr. 1.83) I easily got concurrent results. I had the oil and acid and vessels all at the same initial temperature. The oil was run into a beaker glass of 100 cc capacity, a delicate thermometer was inserted and initial temperature marked and compared with thermometer hanging beside burette containing oil and acid. Then acid was run in and the

* Volume 10, American Public Health Association, reprints for the author an article entitled "The Use of Cotton-Seed Oil as a Food and for Medicinal Purposes."

whole stirred with a glass rod, the lower part being flattened parallel to vertical axis, and stirring continued until the mercury ceased to rise.

Of course the final temperature varied with initial temperature, but the idea was to have samples of standard oils on hand and as the temperature varied in the room from day to day to make comparisons at same temperature between oil under observation and standard oils. Variations also occur with rate of stirring, but it is remarkable how close agreement is with practice. For example I cite—

Experiments made June 18, 1884, with standard oils.

Oil.	Initial temperature.	Final temperature.	Increase in temperature.	Mean.
Lard, winter.....	25.4	64	38.6	39
Do	25	64	39	
Do	25	64	39	
Cottonseed, summer	26	78	52	53
Do	25	79	54	
Do	25	78	53	

Experiments with mixtures of above oils.

Oil.	Initial temperature.	Final temperature.	Increase in temperature.	Increase calculated.	Difference.	Ratio of oils taken.
	°	°	°	°	°	cc
Lard, winter	26	71.0	45	45.9	-0.9	10
Cottonseed, summer.....						10
Lard.....	26	67.4	41.4	42.5	-1.1	15
Cottonseed						5
Lard.....	26	73.5	47.5	49.5	-2.0	5
Cottonseed						15
Lard.....	26	75.5	49.5	49.5	0.0	5
Cottonseed						15
Lard.....	26	69.4	43.4	44.6	-1.2	12
Cottonseed						8
Lard.....	26	75.8	49.8	48.1	+1.7	7
Cottonseed						13
Lard.....	26	76.4	50.4	50.2	-0.2	4
Cottonseed						16

The calculated increase is based on the numbers 39° and 53° given in the first table. It will be observed that the differences reduced to percentages are large, and that the initial temperature of the mixed oils are above those of the original oils, yet there is no question about the detection of the mixture and the estimation of the proportions on a commercial scale. In point of fact what we demanded was pure lard oil only to conform to a certain standard and the test as applied secured this. I have better results than those cited, but this is probably as good as the average inspector will get.

It is said, however, that beech-nut-fed pork gives an oil that yields results like cottonseed. In a thorough study of the subject this should be considered as well as the effects of age and of the different methods of refining.

(f) *Crystallization point of fatty acids.*—The maximum temperature reached during the process of crystallization of the fatty acids is also a

valuable indication. The method pursued in determining this point is as follows: The fatty acids were prepared in sufficient quantities to afford about 50 or 60 grams for analytical purposes. The apparatus used is represented in Fig. 23.

A very delicate thermometer with a long bulb is used, the thermometer being graduated into tenths of a degree; the readings of the mercury are made with a small eye-glass. A test tube about 15 cm in length and 2.5 to 3 cm in diameter is filled with the melted fatty acids. The temperature at which the acid is melted should be sufficiently high to secure a complete liquefaction. The tube containing the fat is placed in a stopper carried in a bottle so that the whole of the fatty acid may be contained in that part of the tube protected from external currents of air by the bottle. The bottom of this protected bottle should be warm, so that its temperature may be several degrees higher than the crystallizing point of the fatty acid. This precaution is necessary to avoid a too rapid crystallization of the fatty acid in the bottom of the test tube and to secure as nearly as possible a uniform crystallization throughout the whole mass. The thermometer is suspended in such a manner that the bulb may occupy as nearly as possible the center of the fatty mass. The thermometer should be protected from currents of air and should be kept perfectly still. The position of the mercury in the thermometer is carefully watched by the attendant as it gradually sinks toward the crystallizing point. When the crystals of the acid begin to appear in the bottom and on the sides of the test tube the descent of the mercury will become very slow and finally cease; the lowest point reached by the mercury should be noted. As the crystallization extends inward toward the bulb of the thermometer a point will be reached when the mercury will begin to rise; at that point, the test tube being held by the left hand, the thermometer should be taken by the right hand of the operator and the partially crystallized mass of fat thoroughly stirred by turning the thermometer three or four times

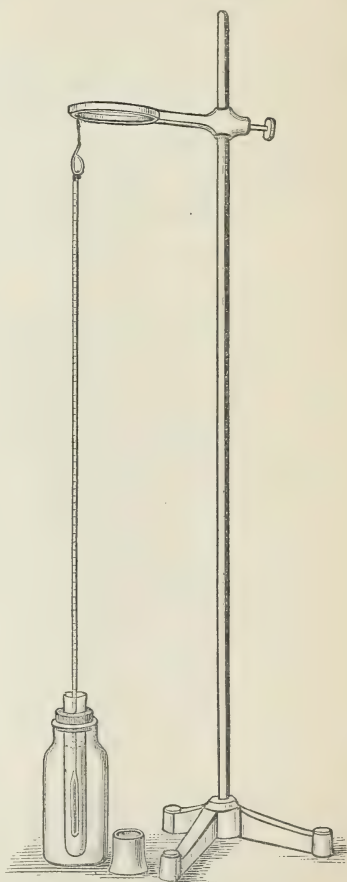


FIG. 23.

around the tube in both directions. Care should be exercised that at the end of this operation the bulb of the thermometer should hang as near as possible in the center of the crystallizing mass. Directly the above operation is accomplished the mercury will be seen to rise and this rise of temperature will continue for some time, after which the mercury will remain stationary for one or two minutes. The highest point reached is taken as the true temperature of crystallization.

(g) *Melting point of the fatty acids.*—The melting point of the fatty acids can not be determined in the same apparatus and by the same methods as those described for the fats themselves, because the acids are soluble in alcohol. It should be remembered that the melting point of the fatty acids is slightly above that of the glycerides, and the first determination in every case should be solely for the purpose of determining approximately the temperature at which the fat melts.

METHOD OF DETERMINING MELTING POINT OF FAT ACIDS.

(1) *By capillary tubes.*—The fat acid in a capillary tube is placed in a beaker of water, together with a delicate thermometer. The water is slowly heated and the point at which the fat becomes transparent is noted.

(2) *In a closed flask.*—This method, easy of application and giving satisfactory results, was proposed by Mr. Oma Carr.

The bulb of a delicate thermometer is coated with the fat acid, and the thermometer, by means of a cork is fastened in a round flask of 250cc capacity. The bulb of the instrument should occupy as nearly as possible the center of the flask. The cork should have an air passage for the equalization of the pressure. The flask is slowly heated in a current of warm air, or otherwise, and as the melting point is approached a rotatory movement is given to it. When the fat melts it is seen to collect in a small drop on the lowest part of the bulb, remaining stationary while the flask is turned. The thermometer is best held horizontally.

(c) *Color reaction.*—The re-agents used in determining the color reactions were sulphuric acid of a specific gravity 1.7 and strong nitric acid. The method of working with sulphuric acid is as follows: A porcelain plate with trough like indentations, such as is used by artists in mixing paints, is employed. The plate is warmed to a temperature slightly above that of the fat to be examined, and inclined slightly so that the liquid fat may remain in the lower end of the trough. A few drops of the fat are placed on the dish, which is capable of holding several samples; a few drops of the sulphuric acid are next placed upon the samples of fat and each one stirred with a short glass rod. The coloration produced is carefully noted, the beginning of the change in color noticed and its progress watched. The samples should also be allowed to remain for twelve hours and the coloration produced at the end of that time studied.

The method of proceeding with nitric acid is as follows :

Small test tubes are taken which are filled one-third full of the melted fat and an equal volume of the strong nitric acid is added, the test tube closed by a piece of rubber cloth, held firmly down by the thumb, and vigorously shaken for a minute. The tube is then placed in a rack and the oily layer allowed to separate from the acid. The oil being lighter rests upon the top of the acid. The coloration produced is studied in the same manner as has been indicated for sulphuric acid.

METHOD OF DETERMINING THE RELATIVE PROPORTIONS OF STEARIC AND OLEIC ACIDS IN A MIXTURE OF THE TWO.

The method proposed by Dalican and Jean rests upon the use of data of temperatures produced by the act of crystallization of the two acids. This point is determined in the manner already described.

The proportions of the two acids are then calculated from the following table :

Temperature of thermometer in degrees C.	Percentage of stearic acid.	Percentage of oleic acid.	Temperature of thermometer in degrees C.	Percentage of stearic acid.	Percentage of oleic acid.
40.....	35.15	59.85	45½.....	52.25	42.75
40½.....	36.10	58.90	46.....	53.20	41.80
41.....	38.00	57.00	46½.....	55.10	39.90
41½.....	38.95	56.05	47.....	57.95	37.05
42.....	39.90	55.10	47½.....	58.90	36.10
42½.....	42.75	52.25	48.....	61.75	33.25
43.....	43.70	51.30	48½.....	66.50	28.50
43½.....	44.65	50.35	49.....	71.25	23.75
44.....	47.50	47.50	49½.....	72.20	22.80
44½.....	49.40	45.60	50.....	75.05	19.95
45.....	51.30	43.70			

For mixtures of acids such as are afforded by the saponification of compound lards, the table appears to be valueless. Many of the temperatures of crystallization of such acids, as can be seen from the tables of analyses, fall below 40°.

Dr. Crampton, to whom I assigned the microscopic examination of the lards and lard compounds, has contributed the following account of the work :

CRYSTALLIZATION OF FATS.

Microscopic examination.—Probably the first application of the use of the microscope for distinguishing between fats derived from different sources was by Husson,* who obtained crystals from beef fat, tallow, lard, oleomargarine, goose fat, butter,

* Jour. de Pharm. et de Chim., 4^e série, vol. 27, p. 100.

etc., by dissolving them in a mixture of alcohol and ether, cooling the solutions, and allowing the fats to crystallize out. He claims that he could distinguish the crystals obtained from different fats in this way, and gives illustrations made from drawings of the various forms of crystals. The delineations are very crude and poor.

In the famous "Chicago lard case"* microscopical methods were employed by experts for the defense to distinguish pure lard from lard adulterated with beef fat. Dr. W. T. Belfield seems to have been the first in that trial to point out the differences between crystals of these fats obtained from their solutions in ether or alcohol, and his methods and conclusions were followed and confirmed by most of the scientific experts employed by the defense. Dr. Belfield claimed to be able to recognize in this way as little as 10 per cent. of beef fat in lard, while some of the other gentlemen thought that as low a proportion as 5 per cent. could be shown. Twelve photomicrographs were submitted by Dr. Belfield as part of his testimony, and are reproduced in the report of the trial. They consist of crystals obtained from pure lard, both steam and kettle rendered, pure tallow, lard mixed with 30, 20, and 10 per cent. of beef tallow, and crystals from the three suspected samples of lard, "Fowler's" Nos. 1, 2, and 3. The reproduction is fairly good, but the amplification is not stated.

In Part I of this Bulletin is presented a discussion of the microscopical appearances of the various fats used in the adulteration of butter, especially the characters presented by them when viewed by polarized light, with photomicrographs prepared by Messrs. Richards & Richardson.† These are intended especially to show the use of the microscope with polarized light in distinguishing butter from its adulterants, the former having, unless it has been melted and cooled or crystallized from solvents, no crystalline structure, hence showing no refracting bodies when viewed by polarized light, while the substitutes, involving as they do in their preparation previous melting and consequent crystallization, show a variegated field. Some of these photomicrographs represent crystals obtained from lard and beef fat by crystallization from ether or alcohol, but the illumination by polarized light does not give the perfect delineation of the shape of the individual crystals necessary for their differentiation.

Examination of lards and lard substitutes with the microscope.—In the microscopical work on the larger series of samples used in the present examination, and the investigation of the efficiency of this test in distinguishing between lard and its substitutes, I had the benefit of the advice and experience of Prof. S. P. Sharpless, one of the chemists employed in the Chicago trial, who has had occasion to examine microscopically a large number of lards and lard substitutes in the course of an extensive commercial experience. Most of the samples were examined by both of us, and our results agreed with very few exceptions. I subjected the entire series of samples to a very careful examination, making several crystallizations in nearly every case, and making photo-micrographs of the appearances found in a large number of the samples, selections from which are reproduced in the plates.

Methods of procuring crystals for examination.—The methods employed by the experts in the Chicago case, in these microscopical examinations, filed as part of their testimony before the Board of Trade, are given on page.

It will be seen that there was considerable diversity in the solvents used, the manner of crystallization, and the method of preparing the crystals for examination.

My method of procedure was similar to those given, in a general way. About 2 to 5 g of the samples were taken, dissolved up in 10 to 20cc of ether in a test tube, the operation being generally hastened by warming, the tube loosely stopped with cotton, and allowed to stand over night at the ordinary temperature of the room. The proper proportions of substance and solvent can not be laid down absolutely as they are dependent upon so many conditions of temperature, solubility of the sample, etc. The proportion giving a proper rate of crystallization, neither too rapid or too slow, can

* McGeech, Everingham & Co. vs. Fowler Brothers, published by Knight & Leonard. Chicago, 1883.

† Pp. 34-40.

best be found by experiment. It will differ with different samples, and with the time of year, unless the temperature of the room in which the crystallization takes place be artificially controlled.

The crystals which have formed at the bottom of the test tube are taken out with a piece of glass tubing, placed on a slide, covered with a cover glass, and examined with a $\frac{1}{2}$ or $\frac{1}{4}$ inch objective. Sometimes the mother-liquor will not be sufficiently concentrated to furnish a medium for the observation of the crystals, the evaporation of the ether leaving them dry; in such cases the addition of a little cotton-seed oil will be found advantageous. I did not find any advantage in washing the crystals obtained with alcohol. I made a number of experiments in the crystallization from different solvents, alcohol, benzol, turpentine, chloroform, etc., but obtained often very different crystals from the same fat crystallized from different solvents.

On the theory that the crystals characteristic of beef fat are composed of stearine, which would probably crystallize out before the other glycerides, if present, some of the experimenters quoted above lay stress upon the examination of the first crystals formed, with the idea that these would be the beef crystals. I have not found such to be the case, the crystals formed when the solution had become concentrated being generally like those first produced, except that they were not so perfect and distinctive, having been more rapidly formed. Nor have I been able often to find such appearances as are shown by Dr. Belfield's plates of mixtures of lard with 20 and 10 per cent. of tallow, and which show the characteristic beef crystals among characteristic lard crystals on the same slide. In only two or three cases did I find the two together on the same field, and I am unable to show a single photograph of such a field, though I endeavored to make such a slide. My experience has been that the kind of crystallization first instituted predetermined the general form of all subsequent crystals.

Slight differences in the temperature or in the concentration of the solution when crystallization began seemed to have an influence upon the form of crystals produced when the substance was a mixed fat, so that in some cases where no beef crystals could be detected in a solution even by examining it at different periods, if another solution were made and allowed to crystallize, beef crystals would appear.

In Plates XXXI and XXXII are shown the characteristic crystals obtained from pure lard* when crystallized from ether; in Plate XXXVIII the crystals from beef. From these it will be seen that these fats, taken separately, give very different crystals. Just what these distinctive crystals are is a most interesting question, both in a theoretical and practical point of view. Some of the experts quoted above evidently thought, from their testimony, that the lard crystal was palmitine, and the beef stearine. Others seem to think they were both stearine modified and that this glyceride crystallizes in different forms in the different fats. Whether these different crystals are really composed of distinct glycerides, or whether they are mixtures of different but definite proportions of the various glycerides found in fat, are questions that can not be answered in the present state of our knowledge. All we can say is that they are quite different in appearance and that pure lard always gives the one, pure beef fat the other form. Not only are the forms of the individual crystals different, but the manner of aggregating themselves together is also quite distinct.

This is seen from Figs. 1 and 2, Plate XXIX, in which a small power was used in order to show the manner of aggregation of the lard crystals. They form feathery masses, radiating from a longitudinal axis, with similar secondary branches. The beef fat crystals on the other hand, as is shown in Plates XXVI and XXVIII, from spherical masses radiating from a common center, breaking up under the cover-glass into fan-

* The lard crystals make rather a difficult subject to photograph; they are very thin and the difference in transparency between them and the field is very little. The slight refraction of light by their edges shows their outline on the plates, but only a very delicate impression is made on account of the very thin edge.

shaped clusters, often with a peculiar twisted appearance. If the individual beef fat crystals are magnified further they still show their needle-like form, but by increasing the amplification of the cluster of lard crystals, shown in Fig. 2, Plate XXIX, for instance, we would get a similar appearance to that of Fig. 4, Plate XXX, or Fig. 9, Plate XXXIII, and by a still higher power the terminations of the crystals are plainly shown as in Fig. 5, Plate XXXI. The differences between the typical crystallization of beef and hog fat are thus easily recognized; if now the mixture of the two fats gave, on crystallizing, a mixture of the different forms in the proportion of the mixture, the recognition of such a mixed fat would be very easy even though the proportion of the one ingredient greatly preponderated. But such has not been my experience; instead of obtaining from a mixture of 10 per cent. beef fat and 90 per cent. lard, for example, a crystallization containing a great many lard crystals with a few beef fat crystals scattered amongst them, as shown by Dr. Belfield, I usually found a uniform kind of crystallization, which varied from either typical form, but which resembled more the lard. Some of these were extremely difficult to identify positively, and I was obliged to recrystallize repeatedly and vary the conditions before I could obtain sufficiently characteristic forms. Take the appearance shown in Fig. 12, Plate XXXIV, for instance: the manner of aggregation is like that of beef fat crystals, but if the individual crystals are examined by a high power, it will be seen that they are not needle-shaped and pointed, but plates with oblique terminations, although not nearly so thin or tabular as the typical lard crystals. Most of Armour's lards presented these difficulties, the appearance shown in the two figures on Plate XXXV being exceptional in this respect, and showing very plain evidence of beef admixture. Most of them gave appearances similar to Fig. 12. In two of Armour's lards, viz, serial Nos. 5557 and 5559, I was unable to find any evidence of beef fat admixture; the crystallization showing always good typical lard crystals. Compound lards from lard and cotton-seed oil only would react in this way. In Fairbank's lards on the other hand, which contain a larger proportion of beef fat, it is often difficult to obtain anything except the beef fat appearance. Pure lards sometimes give appearances, which might be mistaken for beef fat. Fig. 4, Plate XXX, for example, if viewed with a low power might possibly be mistaken for beef fat aggregations, but this mistake need not be made if the terminations of the crystals be carefully examined. The lard crystals when turned up on edge sometimes look like beef fat crystals. In the examination of a sample suspected of being compounded with beef fat, it is the beef fat appearance, of course, that is to be sought for, unless there is some special reason for knowing whether it contains any lard at all, and as soon as a characteristic beef fat crystallization is observed the object of the examination is attained. If none but lard crystals are observed at first, however, it must not be concluded at once that the sample is a pure lard, but the crystallization

must be repeated, and only after a number of recrystallizations have been made, and many slides taken with no appearance of beef fat crystals can it be decided that no beef fat is present. I should say, as a result of my own observations, that as small an admixture as 20 per cent. of beef fat can readily be detected, but I should hesitate very much about guarantying a detection of 10 per cent. or less, as the experts in the Chicago case were confident of doing.

The presence of a large amount of cotton seed oil facilitates, of course, the detection of beef fat admixture by the microscope. That is, a compound lard made up with say 10 per cent. of beef fat stearine, 50 per cent. of lard, and 40 per cent. of cotton seed oil would be more likely to give a characteristic beef fat crystallization than one made up with 10 per cent. stearine, 65 per cent. lard, and 25 per cent. cotton seed oil, for the proportion of lard to beef fat would be greater in the latter case and hence more likely to predetermine a formation resembling the lard crystals. Under the ordinary conditions of crystallization no crystals would likely be obtained from the cotton seed oil. The crystallization shown in Fig. 18, Plate XXXVI, was obtained from a concentrated solution of cotton-seed stearine in ether after it had stood at ordinary temperatures for nearly a week.

DESCRIPTION OF PLATES.

PLATE XXIX.

- FIG. 1. Lard from G. Cassard & Son, Baltimore. Serial No. 5606.
FIG. 2. Lard from intestine of hog rendered in laboratory U. S. Department Agriculture. Serial No. 5673.

PLATE XXX.

- FIG. 3. Lard from G. Cassard & Son, Baltimore. Different crystallization from Fig. 1. Serial No. 5606.
FIG. 4. Prime steam lard from C. H. S. Mixer, inspector, Chicago, Ill. Serial No. 5662.

PLATE XXXI.

- FIG. 5. Leaf lard rendered in laboratory U. S. Department Agriculture. Serial No. 5674.
FIG. 6. Lard from intestine of hog rendered in laboratory U. S. Department Agriculture. Serial No. 5673.

PLATE XXXII.

- FIG. 7. Lard from J. P. Squire & Co., Boston, Mass. Serial No. 5591.
FIG. 8. Same as Fig. 7. Another slide.

PLATE XXXIII.

- FIG. 9. Lard from Jacob Shafer, Baltimore. Serial No. 5550.
FIG. 10. Leaf lard rendered in laboratory. Serial No. 5674.

PLATE XXXIV.

- FIG. 11. Leaf lard rendered in laboratory. Serial No. 5674.
FIG. 12. Refined lard from Armour & Co., Chicago, Ill. Serial No. 5611.

PLATE XXXV.

- FIG. 13. Refined lard made by Armour & Co., Chicago, Ill. Serial No. 5610.
FIG. 14. Same as Fig. 13. Another slide.

PLATE XXXVI.

- FIG. 15. Refined lard made by Fairbank & Co., Chicago, Ill. Serial No. 5569.
FIG. 16. Refined lard made by Fairbank & Co., Chicago, Ill. Serial No. 5574.

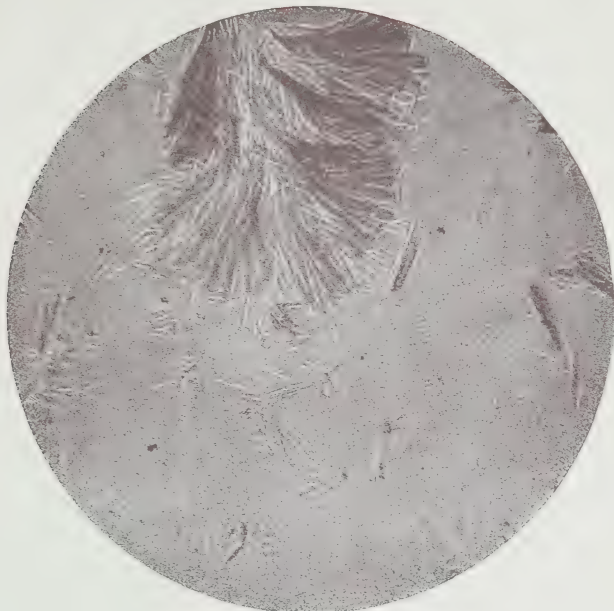
PLATE XXXVII.

- FIG. 17. Lard stearine made by Armour & Co., Chicago, Ill. Serial No. 5613.
FIG. 18. Cotton-seed stearine from Southern Cotton Oil Trust. Serial No. 5680.

PLATE XXXVIII.

- FIG. 19. Oleostearine from N. K. Fairbank, Chicago, Ill. Serial No. 5644.
FIG. 20. Same as Fig. 19. Another slide.

Fig 1



CASSARD'S LARD x 30

Fig 2



INTESTINAL LARD x 30

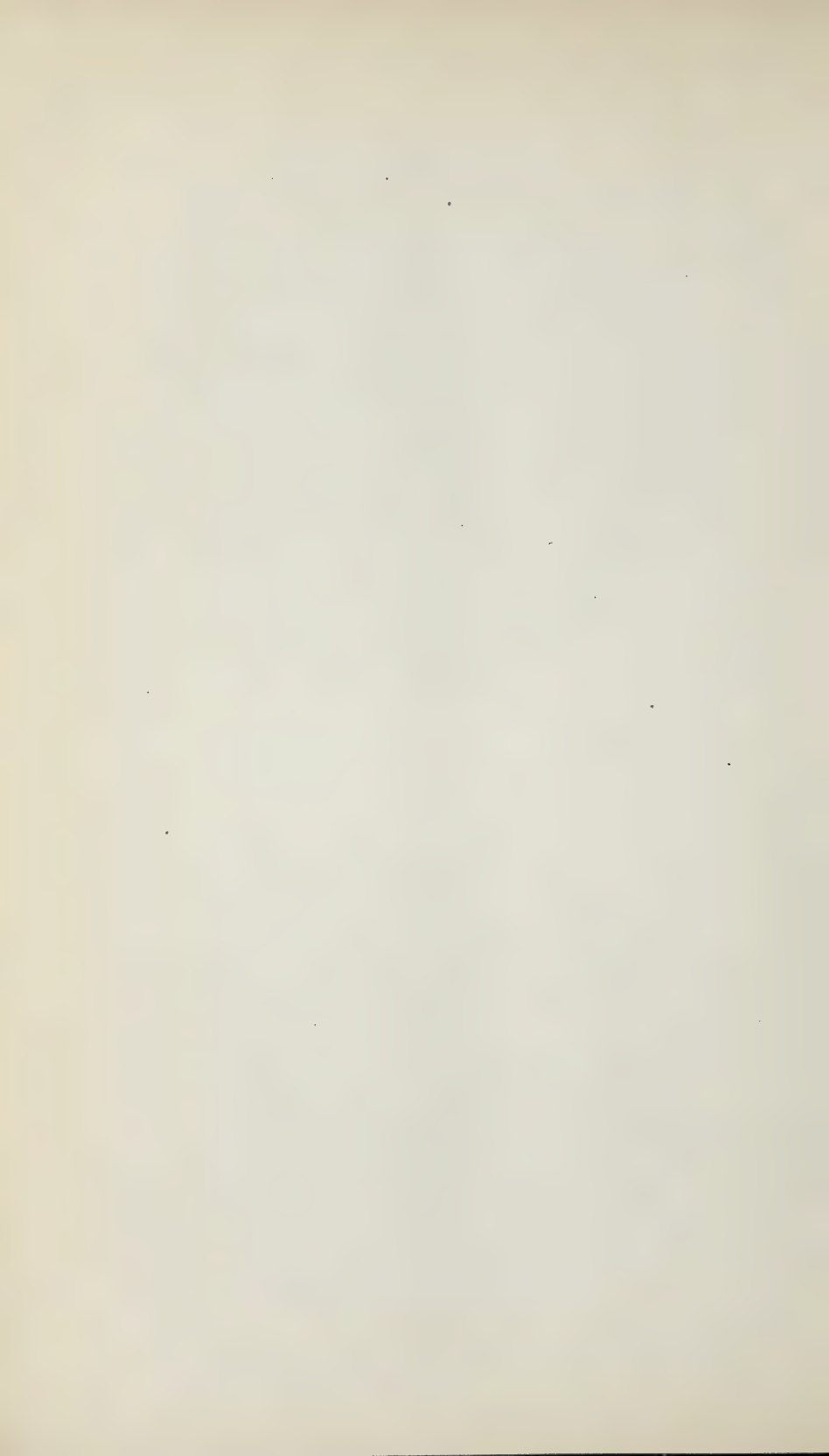
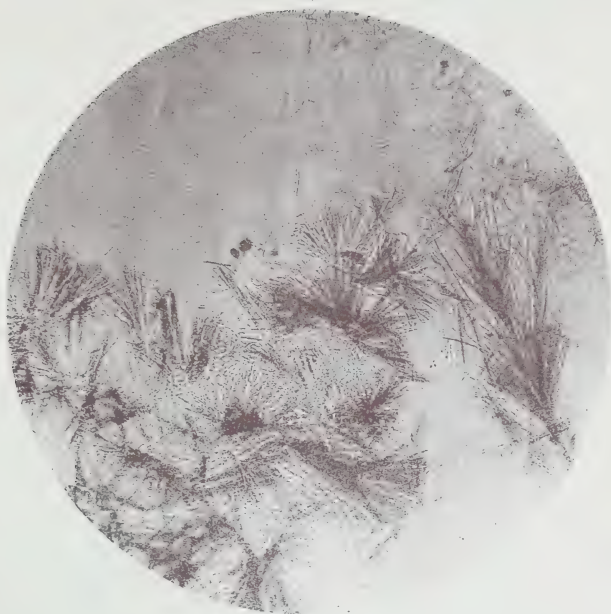


Fig 3



CASSARD'S LARD x30

Fig 4



PRIME STEAM LARD x65



Fig 5



LEAF LARD x185

Fig 6



INTESTINAL LARD x65

Fig 7



SQUIRE'S LARD x65

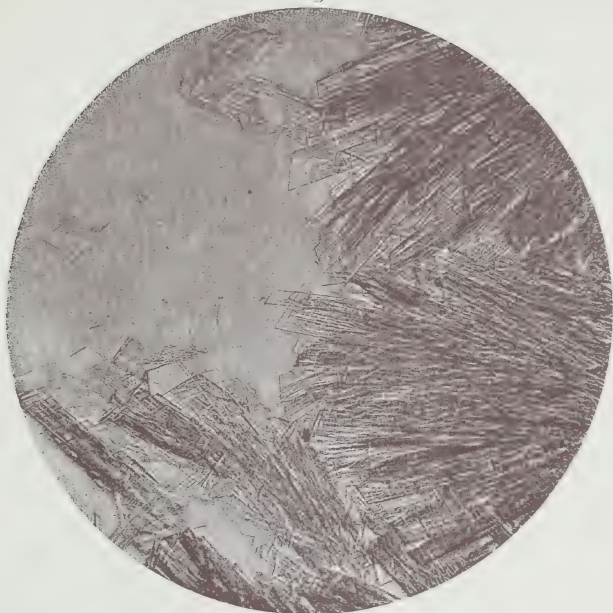
Fig 8



SQUIRE'S LARD x65



Fig 9



SHAFER'S LARD x65

Fig 10



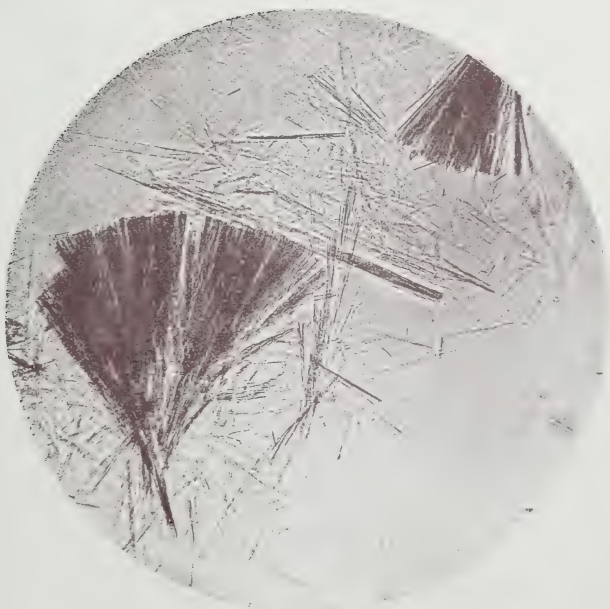
LEAF LARD x65

FIG 11



LEAF LARD x65

FIG 12



ARMOUR'S REFINED LARD x65

Fig 13



ARMOUR'S REFINED LARD x65

Fig 14



ARMOUR'S REFINED LARD x65

Fig 15



FAIRBANKS' REFINED LARD x65

Fig 16



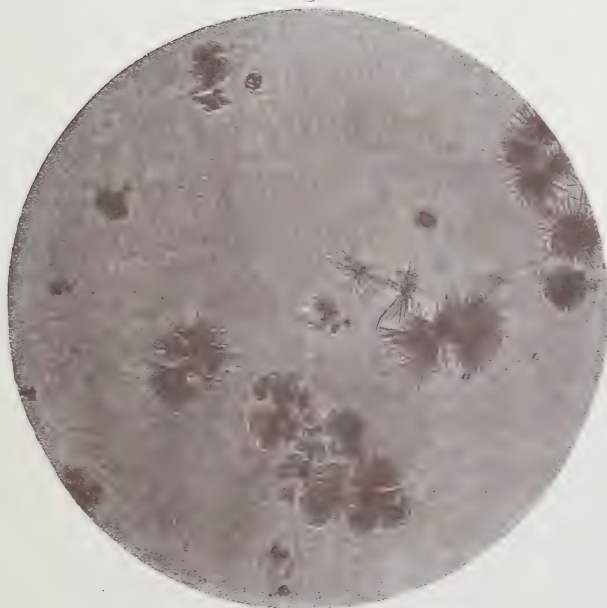
FAIRBANKS' REFINED LARD x65

Fig 17



PRIME LARD STEARINE x65

Fig 18



COTTON OIL STEARINE x65



Fig 19



FAIRBANKS' OLEO-STEARINE $\times 65$

Fig 20



FAIRBANKS' OLEO-STEARINE $\times 65$



B.—CHEMICAL PROPERTIES.

(a) *Volatile or soluble and insoluble acids.*—The determination of the volatile acid is made in the apparatus represented in Fig. 24. The soluble acid may be estimated by the process described in Bulletin No. 16, a résumé of which follows.*

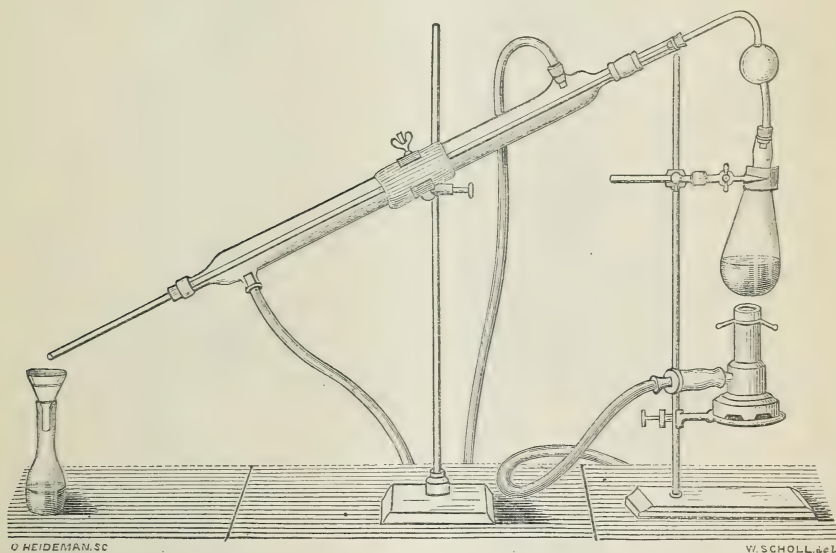


FIG. 24.

METHOD FOR THE DETERMINATION OF SOLUBLE AND INSOLUBLE ACIDS.

Reagents.—(1) A standard semi-normal hydrochloric-acid solution, accurately prepared.

(2) A standard deci-normal soda solution, accurately prepared; each 1 cc. contains .0040 grams of NaOH and neutralizes .0088 grams of butyric acid, $C_4H_8O_2$.

(3) An approximately semi-normal alcoholic potash. Dissolve 40 grams of good stick potash in 1 liter of 95 per cent. alcohol, redistilled. The solution must be clear and the KOH free from carbonates.

(4) A 1 per cent. solution of phenolphthalein in 95 per cent. alcohol. Saponification is carried out in rubber-stoppered beer bottles holding about 250 cc, or in a round-bottom strong flask used in distillation.

About 5 grams of the melted butter fat, filtered and freed from water and salt, are weighed out by means of a small pipette and beaker, which

* Bulletin No. 16, page 70.

are reweighed after the sample has been taken out and run into a saponification bottle; 50 cc of the semi-normal potash are added, the bottle closed and placed in the steam-bath until the contents are entirely saponified, facilitating the operation by occasional agitation. The alcoholic potash is measured always in the same pipette, and uniformity further insured by always allowing it to drain the same length of time, viz, thirty seconds. Two or three blanks are also measured out at the same time and treated in the same way.

In from five to thirty minutes, according to the nature of the fat, the liquid will appear perfectly homogeneous, and when this is the case the saponification is complete, and the bottle may be removed and cooled. When sufficiently cool the stopper is removed and the contents of the flask rinsed with a little 95 per cent. alcohol into an Erlenmeyer flask of about 200 cc capacity, which is placed on the steam-bath, together with the blanks, until the alcohol has evaporated.

Titrate the blanks with semi-normal HCl, using phenolphthalein as an indicator. Then run into each of the flasks containing the fat acids 1 cc. more semi-normal HCl than is required to neutralize the potash in blanks. The flask is then connected with a condensing tube 3 feet long, made of small glass tubing, and placed on the steam-bath until the separated fatty acids form a clear stratum on the surface of the liquid. The flask and contents are then allowed to become thoroughly cold, ice-water being used for cooling.

The fatty acids having quite solidified, the contents of the flask are filtered through a dry filter paper into a liter flask, care being taken not to break the cake. Two hundred to three hundred cubic centimeters of hot water is next poured on the contents of the flask, the cork with its condenser tube re-inserted, and heated on the steam-bath until the cake of acids is thoroughly melted, the flask being occasionally agitated with a circular motion, so that none of its contents are brought on the cork. When the fatty acids have again separated as an oily layer, the flask and its contents are cooled in ice-water, and the liquid filtered through the same filter into the same liter flask. This treatment with hot water, followed by cooling and filtration of the wash-water, is repeated three times, the washings being added to the first filtrate. The mixed washings and filtrate are next made up to 1 liter, and 100 cc, in duplicate, are taken and titrated with deci-normal NaOH. The volume required is calculated to the liquid. The number so obtained represents the measure of deci-normal NaOH neutralized by the soluble fatty acids of the butter fat taken, plus that corresponding to the excess of the standard acid used, viz, 1 cc. The amount of soda employed for the neutralization is to be diminished, for the 1 liter, by 5 cc, corresponding to the excess of 1 cc $\frac{1}{2}$ N. acid.

This corrected volume, multiplied by the factor .0088, gives the butyric acid in the weight of butter fat employed. (See table.)

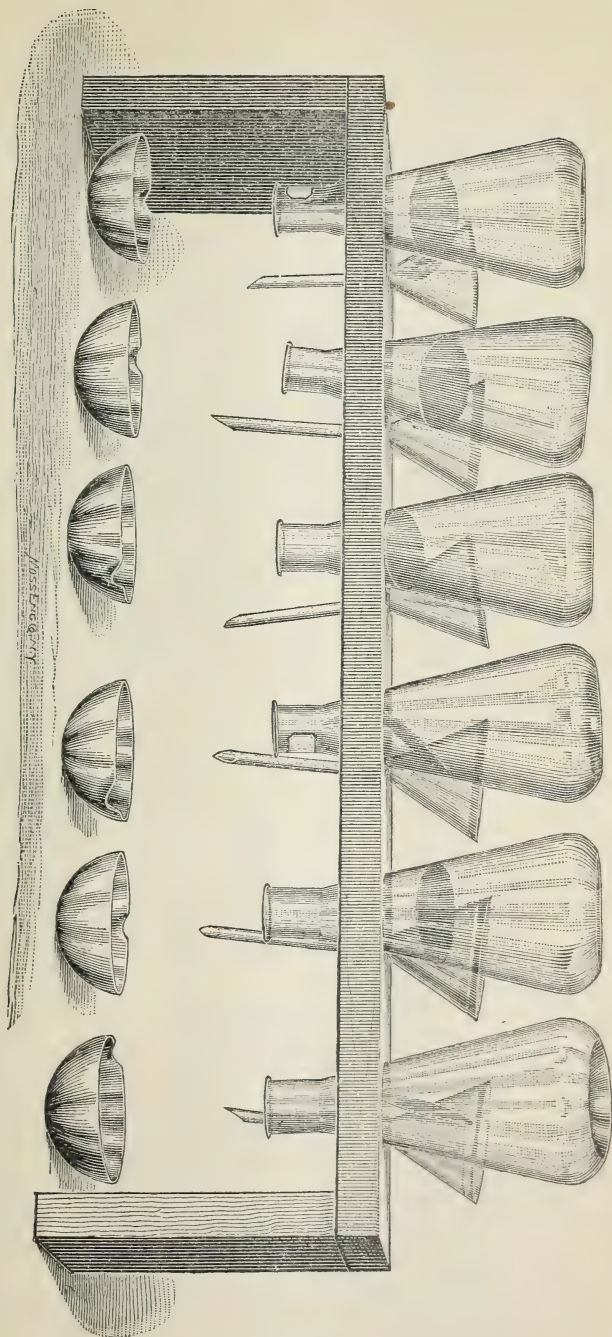


FIG 24 bis.

The flask containing the cake of insoluble fat acids is inverted and allowed to drain and dry for twelve hours (Fig. 24 bis), together with the filter paper through which its soluble fatty acids have been filtered. When dry the cake is broken up and transferred to a weighed glass evaporating dish. Remove from the dried filter paper as much of the adhering fat acids as possible and then add them to the contents of the dish. The funnel, with the filter paper, is then placed in an Erlenmeyer flask, a hole is made in the bottom of the filter paper, and it is thoroughly washed with absolute alcohol from a wash bottle. The flask is rinsed with the washings from the filter paper and pure alcohol, and these transferred to the evaporating dish. The dish is placed on the steam-bath and the alcohol driven off. It is then transferred to the air bath and dried at 100° C. for two hours, taken out, cooled in a desiccator, and weighed. It is then again placed in the air-bath and dried for another two hours, cooled as before, and weighed. If there is no considerable decrease in weight the first weight will do; otherwise, re-heat two hours and weigh. This gives the weight of insoluble fat acids in the quantity taken, from which the percentage is easily calculated.

Table for the calculation of soluble fatty acids.

No. cc KOH Sol.	Equiva- lent.	N 10 NaOH.	Equiva- lent.	N 10 NaOH.	Equiva- lent.	N 10 NaOH.	Equiva- lent.
	<i>Grams.</i>		<i>Grams.</i>		<i>Grams.</i>		<i>Grams.</i>
10.....	.0880	+.25	.0902	+.50	.0924	+.75	.0946
11.....	.0968	25	.0990	50	.1012	75	.1034
12.....	.1036	25	.1078	50	.1100	75	.1122
13.....	.1144	25	.1166	50	.1188	75	.1210
14.....	.1232	25	.1254	50	.1276	75	.1298
15.....	.1320	25	.1342	50	.1364	75	.1386
16.....	.1408	25	.1430	50	.1452	75	.1474
17.....	.1496	25	.1518	50	.1540	75	.1562
18.....	.1584	25	.1606	50	.1628	75	.1650
19.....	.1672	25	.1694	50	.1716	75	.1738
20.....	.1760	25	.1782	50	.1804	75	.1826
21.....	.1848	25	.1870	50	.1892	75	.1914
22.....	.1936	25	.1958	50	.1980	75	.2002
23.....	.2024	25	.2046	50	.2068	75	.2090
24.....	.2112	25	.2134	50	.2156	75	.2178
25.....	.2200	25	.2222	50	.2244	75	.2266
26.....	.2288	25	.2310	50	.2332	75	.2354
27.....	.2376	25	.2398	50	.2420	75	.2442
28.....	.2464	25	.2486	50	.2508	75	.2530
29.....	.2552	25	.2574	50	.2596	75	.2618
30.....	.2640	25	.2662	50	.2684	75	.2706

The table gives the weight of soluble acids (butyric, etc.) for each quarter of a cubic centimeter of deci-normal alkali from 10 to 30.

Example.

Weight fat taken	Grams.
No. cc $\frac{N}{10}$ alkali used	4.967
Less 5cc due to 1cc $\frac{N}{2}$ acid	25.50
Weight soluble fat acids	20.50
Per cent. soluble fat acids1804
	3.63

The modification introduced into the above method is in making the flask in which the saponification takes place and from which the distillation is made the same. For this purpose a specially-made flask such as is used in the digestion in the Kjeldahl method of determining nitrogen is employed. This flask is made of extra heavy glass, well annealed and quite heavy, so as to resist the pressure of the tension of the alcohol at the temperature of the steam-bath. The sample of fat with the saponifying re-agents having been placed in the flask, a stopper of soft cork is inserted and tied down with a string or wire as represented in Fig. 25. The flask is then placed upon a steam-bath and heated for one hour, at the end of which time the fats will be found saponified and any ether which may have been developed decomposed by the excess of alkali present. After cooling the stopper of the flask is removed, the alcohol evaporated, and the decomposing acid added, and the distillation carried on essentially in the manner described.

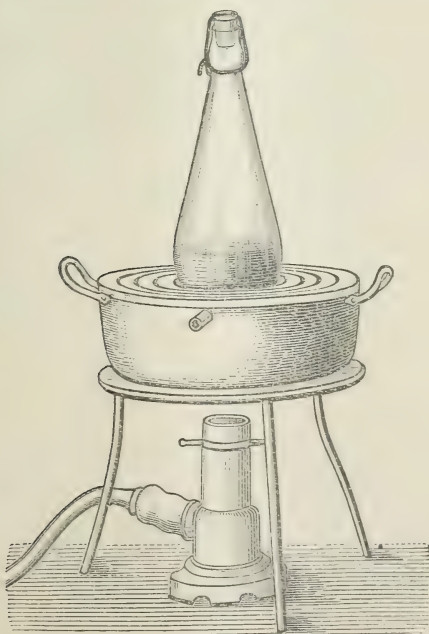


Fig. 25.

This method of procedure avoids the possibility of any loss which might ensue in transferring the saponified fats from the vessel in which the saponification took place into the distilling flask.

In evaporating the alcohol the residual soap sometimes froths and fills the flask. This is avoided by removing the flask from the steam-bath when signs of frothing are shown and rolling it in such a manner as to coat the bottom and lower fourth of the flask with a film of soap. The flask should also be inverted and waved to and fro towards the end of the evaporation in order to remove the vapor of alcohol.

The method proposed by Wollny* has also been used in the estimation of volatile acids. The method is as follows:

Five grams of the fat are weighed into an Erlenmeyer flask; 10cc of alcohol at 95 per cent. and 2cc of concentrated soda lye at 50 per cent., which has been preserved in an atmosphere free of carbonic acid, are added. The flask, furnished with a reflux condenser, is heated, with occasional shaking, in a boiling water-bath for one-quarter of an hour. The alcohol is then distilled off by allowing the flask to remain for three-quarters of an hour in a boiling-water bath. One hundred cubic centimeters of recently-boiled distilled water are then added and allowed to remain in the water until the soap is dissolved. The soap solution is then immediately decomposed with 40cc of dilute sulphuric acid (25cc sulphuric acid to 1 liter), and the flask immediately connected with the condenser. This connection is made by means of a 7mm diameter glass tube, which, 1cm above the cork, is blown into a bulb 2cm in diameter; the glass tube is carried obliquely upwards about 6cm and then bent obliquely downwards; it is connected with the condenser by a not too short rubber tube. The flask is warmed by a small flame until the insoluble acids are melted to a clear transparent liquid. The flame is then turned on with such strength that within half an hour exactly 110cc are distilled off. One hundred cubic centimeters of the distillate are filtered off, placed in a beaker glass, 1cc of phenolphthalein solution added and titrated with tenth normal barium hydrate solution when the red color is shown the contents of the beaker glass are poured back into the measuring glass in which the 100cc were measured, again poured back into the beaker, and again titrated with the barium solution until the red color becomes permanent. The distillation should take place in as nearly thirty minutes as possible.

GENERAL DIRECTIONS FOR WEIGHING THE FATS FOR THE ABOVE DETERMINATION.

The difficulty of measuring exactly 5 grams, as indicated above, is considerable. Since the specific gravity of a fat at any given temperature, say 35° or 40°, is accurately known, I find it more convenient to measure out into the flask a volume of the melted fat which will weigh approximately 5 grams. This can be conveniently done by a graduated pipette, which should previously be warmed to a temperature slightly above that of the melted fat with which it is to be used. Let the specific gravity of the fat to be used at the temperature of measure-

* Milch Zeitung, No. 25, 1888.

ment be .903, then the number of cubic centimeters required to weigh 5 grams would be $5 \div .903 = 5.54\text{cc}$. In a case of that kind, therefore, 5.6cc of the fat should be measured into the flask and its weight accurately determined.

Certain precautions are necessary in weighing the samples of fat in order to secure uniform results. Since the temperature at which the fat is manipulated must be kept approximately at from 35° to 40° , the method of weighing from a weighing-bottle is objectionable. Not only is it difficult to gauge the amount poured out from the weighing-bottle, but the falling temperature influences considerably successive weights. The samples should therefore be weighed in the flasks in which the saponification is to take place. In case this is done in an Erlenmeyer flask, it can be placed directly upon the pan of the balance. If the round-bottom flasks are used, however, they may either be held in a light beaker glass on the pan of the balance or suspended from a hook of the balance by a linen thread. The flasks in which the weighings are to be made should not be wiped with a towel or silk handkerchief within fifteen minutes of the time the weight is taken. It is best in weighing these flasks to remove the desiccating material from the interior of the balance, so as to avoid changes in the amount of moisture deposited on the sides of the flask during the time the weighing takes place. The flask should stand in or near the balance for not less than fifteen minutes before the weighing is made. The flask should be counterpoised on the weight-pan of the balance by a duplicate flask treated in the same way. The empty flask having been weighed, it is removed from the balance and a measured quantity of the fat run into it from a graduated pipette. The flask is now replaced upon the pan of the balance or suspended from a hook by a linen thread, as before described. The reweighing of the flask should not take place before five minutes, so that the fat may have time to cool.

<i>Example.</i>		Grams.
Weight of flask counterbalanced.....		4.3611
Weight of flask plus fat		9.3711
Weight of fat.....		5.0100

In the above case the weight of fat which was required was 5 grams, and the amount as measured, as seen by the above results, was almost exactly that required. At the end of the operation the results can be calculated to exactly 5 grams by simple proportion.

(b) *Saponification equivalent*.—About 2.5 grams fat (filtered and free from water) are weighed into a patent rubber-stoppered bottle or flask, as described above, and 25cc approximately semi-normal alcoholic potash added. The exact amount taken is determined by weighing a small pipette with the beaker of fat, running the fat into the bottle from the pipette, and weighing beaker and pipette again, or the method described above may be used. The alcoholic potash is measured always in the same pipette, and uniformity further insured by always allowing it to

drain the same length of time (thirty seconds). The bottle is then placed in the steam-bath, together with a blank, containing no fat. After saponification is complete and the bottles cooled, the contents are titrated with accurately semi-normal hydrochloric acid, using phenolphthalein as an indicator. The number of cubic centimeters of the acid used for the sample deducted from the number required for the blank gives the number of cubic centimeters which combines with the fat, and the saponification equivalent is calculated by the following formula, in which W equals the weight of fat taken in milligrams and N the number of cubic centimeters which have combined with the fat.

$$\text{Sap. equiv.} = \frac{2 W}{N}$$

If it is desirable to express the number of milligrams of potash for each gram of fat employed, it can be done by dividing 5,610 by the saponification equivalent and multiplying the quotient by 10.

PRESERVATION OF THE REAGENTS USED IN DETERMINING THE VOLATILE ACIDS AND SAPONIFICATION EQUIVALENTS.

In order to secure uniformity of strength in the deci-normal and approximately semi-normal alkali solution employed in the above operations, it is necessary that they be preserved out of contact with the carbonic acid in the air. This is best done by the apparatus used for supplying burettes. In the U tube of this apparatus is placed some of the solution which is to be preserved in the flask itself. The air, therefore, which enters the bottle as the solutions are withdrawn is entirely deprived of carbonic acid by passing through the tube.

(c) *Iodine number—Reagents.*—Twenty-five grams of pure iodine dissolved in 500cc of strong alcohol. Thirty grams of mercuric chloride dissolved in 500cc of strong alcohol.

The solution of mercuric chloride is to be poured into the iodine solution. The iodine solution undergoes a constant change, by which its percentage of free iodine is diminished. This has been ascribed to the presence of impurities in the alcohol, but is doubtless due to a conversion of the iodine into hydroiodic acid and to the disturbing influence of the chloroform used in the subsequent process. There are, however, local changes in the strength of the iodine solution which are noticed from day to day, as is indicated in the examples which follow, the iodine solution being apparently stronger some days than others. These local variations may also be ascribed to the influence of the chloroform on the iodine solution. It is therefore of the utmost importance that the blank titrations in which the strength of the iodine solution is determined should be made on the measured portion of the solution, treated with the same amount of chloroform, and allowed to stand the same length of time as the samples containing the oils or fats whose iodine numbers are to be determined. By this method, although the strength of the iodine solution may appear to vary from day to day, yet this variation will take place *pari passu* with the change in the strength of the iodine

solution in contact with the oils under examination. The effect therefore of this change will not be felt upon the number which expresses the percentage of iodine absorbed.

As an illustration of the progressive change in the strength of the iodine solution the following examples are given. In each case the strength was determined by titration with a deci-normal thiosulphate of soda solution:

A solution of iodine, made up as indicated, showed the following strength on the dates indicated:

Date.	Iodine solution cc.		Thiosulphate solution. cc.
April 5, 1888 ..	10	=	18.2
April 11, 1888 ..	10	=	17.8
April 13, 1888 ..	10	=	19.0
April 14, 1888 ..	10	=	18.3
April 16, 1888 ..	10	=	18.4
April 17, 1888 ..	10	=	18.0
April 19, 1888 ..	10	=	19.4
April 20, 1888 ..	10	=	18.5
April 23, 1888 ..	10	=	17.9

The iodine solution was now allowed to stand in a thick green glass bottle until the 10th of November, 1888. On that date it was found that 10cc of the solution of iodine required only 7.3cc of the solution of thiosulphate soda to neutralize it. It is thus seen that during that time two-thirds of the iodine had disappeared.

*Deci-normal solution of thiosulphate of soda (hyposulphite of soda).—*Reduce to a fine powder about 30 grams of the purest recrystallized thiosulphate of soda; spread this salt in a thin layer over a clean white blotting pad; cover with another pad and subject to pressure. After two or three minutes remove the pad, pour the powdered salt into a dish, and repeat the drying operation. It is better to put the salt all into a dish and respread therefrom on the blotting-pad than to stir the salt on the pad with a spatula. By this latter method some fibers of paper may be mixed with the salt. Weigh exactly 24.8 grams of the dried salt and make up to one liter, at the temperature at which the titrations are made, with recently-boiled distilled water. Since the pure salt is used the solution will be exactly deci-normal. Its strength, however, may be set by weighed portions of resublimed iodine, of which about 1 gram, weighed from a weighing bottle, should be taken for each determination. The solution of hyposulphite of sodium may also be set in the following way: Dissolve 3.874 grams chemically pure bichromate of potassium in distilled water and make the volume up to one liter. Place 20cc of this solution in a glass-stepped flask to which has been added 10cc of a 10 per cent. iodide of potassium solution and 5cc of strong hydrochloric acid. Allow the thiosulphate of sodium solution to flow into the flask from a burette until the yellow color of the liquid has almost disappeared. Add a few drops of starch paste, and with constant shaking continue to add the thiosulphate solution until the blue color just disappears. The number of centimeters

of thiosulphate solution used multiplied by 5 is equivalent to 1 gram of iodine.

Iodide of potassium solution.—One part of iodide of potassium in ten parts of water.

Starch paste.—One gram of starch in fine powder suspended in 100 parts of water and heated to the boiling-point. The paste must be cooled to the temperature of the room before using.

Manipulation.—The quantity of fat to be used is determined by its nature. If it consist largely of cotton oil one-half gram is sufficient. If it be mostly pure lard, one gram may be taken. A measured quantity of the fat corresponding to the weight desired is run into a recently-weighed glass-stoppered flask, and after a few minutes the weight of the flask and oil taken with precautions already noted. The fat is now dissolved in 10cc of chloroform; from 20 to 30cc of the iodine solution are then added from a burette. If the solution is not perfectly clear, more chloroform should be added. The amount of iodine employed should be large enough to leave an excess of 8 or 10cc unabsorbed at the end of the reaction. It is important, to secure comparative results, to have the amount of iodine in excess in each case approximately the same. This can be easily secured by a preliminary determination of the approximate amount of iodine absorbed. At the same time two blank determinations are made to determine the strength of the iodine solution; the manipulation in all cases being the same as in those samples containing the fat, save that the fat is omitted. After standing for two hours from 10 to 20cc of the iodide potassium solution are added and 150cc of distilled water, and the liquids thoroughly shaken together. The decinormal solution of thiosulphate of soda is added until the yellow color of the liquid has almost disappeared. The titration is continued after the addition of a few drops of starch paste until the blue color has entirely disappeared.

	<i>Example.</i>	Grams.
Weight of flask		74.1288
Weight of flask, plus fat.....		74.8168
Weight of fat.....		.6880

After the addition of 10cc of chloroform and 30cc iodine solution the flask was allowed to stand two hours. Twenty cubic centimeters of the iodide solution were then added and 150cc of water, and the titration made with deci-normal thiosulphate solution, using starch paste as indicator. The amount of thiosulphate used was 21.2cc. The strength of the iodine solution determined by blank experiment was 10cc of the iodine solution=19.4cc of thiosulphate solution. Since 30cc of the iodine solution were used the amount of deci-normal thiosulphate solution necessary to combine with the whole of the iodine would be 58.2cc; then $58.2\text{cc} - 21.2\text{cc} = 37\text{cc}$, the number of cubic centimeters equivalent to the iodine absorbed by the fat. In a deci-normal thiosulphate solution each cubic centimeter equals .0127 grams of iodine; the total amount of iodine absorbed therefore was $37 \times .0127 = .4690\text{g}$. Then the percentage of iodine absorbed = $.4699 \times 100 \div .688 = 68.29$,

To avoid the disturbing effect of the chloroform in the above process Mr. A. H. Allen recommends the use of the fat acids for absorbing iodine instead of the natural glycerides.

(d) *Reaction with nitrate of silver.*—* The solutions used have been of two kinds, viz: (a) one-fifteenth to one-tenth gram of Ag NO_3 in 200cc of 95° alcohol and 20cc ether.

Of this solution 10cc should be taken for each test. (b) One gram Ag NO_3 in 200cc of equal parts of alcohol and ether. Of this solution 1cc was used. The mixture of 85 parts of amyl alcohol and 15 parts of rape-seed oil was the same in both cases, 10cc of the mixture being used in each test. The method of making the test has also been changed. I use a porcelain dish 8 to 10cm in diameter. The re-agents with the oil (10cc) are thoroughly mixed by shaking in a test tube and then poured into the dish and placed on a steam bath. The contents of the dish are occasionally stirred and the heating is continued for twenty minutes. The deposition of silver on the dish is easily seen and the resulting colors show more clearly on the white porcelain.

Solution (b) acts more promptly than (a), but the results with (a) are more satisfactory.

The order of the phenomena will be found to be as follows:

For pure cotton oils.—In from two minutes to three minutes the mixture turns red. In five to ten minutes the red color becomes so brown as to appear black, in thick layers. At the end of the test metallic silver is deposited on the sides of the dish varying in color from bluish black to reddish purple. The liquid carries also particles of reduced silver and has a decided greenish tint.

With lards containing more than 20 per cent. of cotton oil the phenomena observed above are repeated, but not so promptly.

Even with very small percentages of cotton oil, the characteristic reactions are given.

Animal fats give no color, under similar treatment, or at most a faint red after twenty minutes.

Of vegetable oils I have examined rape seed, olive, peanut, and linseed. These act with the re-agent like the animal fats.

One hundred samples of lard and twenty-five samples of cotton oil have been examined by Bechi's test. In no case, where cotton-seed oil has been present, has the test failed in detecting it, except in two doubtful cases of alleged cotton oil to be mentioned further on.

Of the 100 samples of lard examined 74 were found to be adulterated with cotton oil.

SOME PECULIAR REACTIONS.

The reaction with crude cotton oil is not as sharp as with the refined oil. The deep red color of the sample seems to obscure the final color reactions.

Linseed oil gave a reddish color but no reduction of silver. In three

* Journal of Analytical Chemistry, Vol. 2, part 3, July, 1888.

samples of lard made by us from the leaf, guts, and head, respectively, of the same hog, the re-agent acted in the same way. There was a slightly greater coloration with the head and gut lard than with the leaf.

In samples of "prime steam lard" passed by the Chicago Board of Trade and made from the trimmings of the whole animal, not presumably including the leaf, there-agent gave, after twenty minutes, a slight brownish red color, but no appreciable reduction.

In the whole number of examinations made there were three or four cases in which the results appeared doubtful. A slight reduction of the silver was observed and a color approaching a brown-black, but not with sufficient positiveness to prove the presence of cotton oil. These I have included under the adulterated samples.

In general, it may be said that any degree of adulteration of lard with cotton oil which would prove commercially profitable is at once detected with certainty by Bechi's test.

On the other hand very impure lards containing no cotton oil will give color reactions and a trace of reduction of metallic silver with Bechi's re-agents similar but not identical with a trace of cotton oil.

The reaction is undoubtedly the most valuable single test for cotton oil which has been proposed.

It remains to be seen what reactions lard which is made from swine fattened on mast or cotton meal will give with these re-agents. The nature of the reducing agent has not yet been determined. It has been suggested that it is an aldehyde. It appears to withstand saponification, and Milliau has lately proposed to use the test on the free, fatty acids of cotton oil.

Since cotton oil is sometimes refined with alkaline substances, and thus retains an alkaline reaction, it may happen in the application of the above test that a sufficient amount of alkali is present to reduce the nitrate of silver to oxide. In such a case the proper reaction of the cotton oil may be wholly obscured. To avoid this it is best to make the solution of nitrate of silver distinctly acid by the addition of a small quantity of pure nitric acid.

MILLIAU'S METHOD OF APPLYING THE NITRATE OF SILVER TEST.

The method of Milliau differs from that of Bechi in applying the solution of silver nitrate to the fat acids instead of to the original glycerides. The saponification may be made in any of the usual ways. About 5cc of the fat acids are sufficient for making the test, which is carried on in a test tube 12cc in length and 3cc in diameter. To the acid are added 20cc of strong alcohol and heat applied until the fat acids are dissolved. Add 2cc of a silver nitrate solution containing 30 grams of silver nitrate in 100cc of water; heat in steam bath until about one-third of the alcohol is evaporated. At the end of this time, if the samples be cotton oil or contain cotton oil the silver will be reduced to a metallic state, producing a brown or black color in the

liquid, or give particles of reduced silver in the liquid or on the sides of the tube.*

I have employed the following modification of Milliau's method, which acts more satisfactorily than the original.

The re-agents are placed in a round-bottomed porcelain dish of about 50cc capacity. The silver re-agent is acidified by the addition of from .5 to 1cc of pure nitric acid. The reaction is conducted on a steam bath.

With the fat acids of cotton oil the order of phenomena is as follows:

The fat acid being thoroughly dissolved and warmed to the evaporating point of the alcohol, 2cc of silver re-agent are added and quickly stirred in with a glass rod. An almost immediate deep brown coloration is noticed, passing quickly to black. As the alcohol evaporates, the reduced silver collects in mirror-like scales and is carried onto the sides of the dish by the escaping alcohol. In a few minutes the liquid begins to grow clear again, and in ten minutes almost the whole of the reduced silver is attached to the sides of the dish.

The fatty acids for use in this modification were separated in the summer of 1888, but on account of a stress of other duties the work was not done until in December. The notes of these tests were mislaid, and in February, 1889, the work was again done.

The results of the second set of determinations were quite surprising, and lead one to suppose that the fat acids should not be kept a long while before treatment with the silver solution.†

In most cases the reactions were quick and satisfactory, but in a few cases entirely misleading. The fat acids of some cotton oils failed to give any reductions whatever, and in some of the mixed lards, where cotton-oil was known to be present, the reduction was so slight as to be wholly useless for analytical purposes. My experience with more recently-prepared samples showed that in such cases the anomalies mentioned above are not repeated. With pure lards there was also a trace of reduction noticed in some cases, which I suppose would not be seen in the freshly-prepared samples. The reaction with cottonseed oil acids, when it appeared at all was so clear and unmistakable, as to lead me to believe that in these respects the process of Milliau is an improvement on the method of Beechi. In the instances marked "trace of reduction" the separation of a slight amount of a substance was noticed, which, however, was usually of a brown color, and did not resemble in any marked degree the intense blue-black mirror-like deposit of silver by pure cotton oil. The use of the terms *trace of reduction* and *slight reduction* in the tables should not be construed into evidence of the presence of cotton-oil fat acids in the samples so marked.

The *marked* and heavy *reductions* were attended by an immediate brown color on adding the re-agent, passing rapidly into black. After heating for a few minutes the silver was deposited as black mirror-like

* Journal of the Chemical Society, August 31, 1888.

† This loss of reducing power in samples of cotton oil long kept has also been noticed by other observers.

particles on the sides of the dish and the liquid became almost colorless again.

WARREN'S CHLORIDE OF SULPHUR TEST.

The method of investigation employed was the one described by Warren.* It is as follows:‡

Five grams of the oil or mixture are weighed in a tared porcelain dish, which is well glazed both inside and out; it should have a capacity of about 4 ounces, so as to avoid loss from spitting. It should not be covered. Two cubic centimeters carbon disulphide are stirred in and 2cc of the mixture of sulphur chloride added. It is now placed on a hot water bath and well stirred until the action is fairly commenced; when solidified it is placed in a warm chamber, so as to drive off all volatile products. When two successive weighings are the same, it is ready for further operation. The mass will require breaking up, so as to allow imprisoned vapors to escape.

The color and consistency at the end of the reaction and when subsequently dried should be noticed; it is now ground up or divided as much as possible. The product may be too tough to break easily, or, if soft and sticky, a portion of the unaltered oil should be removed first.

It is transferred to a filter tube and washed with carbon disulphide, so as to remove all traces of unaltered oil, etc., which is received in a tared flask; about 200cc will suffice in any case. It is best to break up the mass after a partial exhaustion, especially when the product is hard and tough or soft and adhesive.

Oils, fats, resins, rosin oils, petroleum, etc., not acted on by sulphur chloride so as to yield solid products, may be separated. The melting point of a fat before and after separation of the oil is an interesting and useful matter. The viscosity of a mixture containing an ingredient acted on by sulphur chloride is of importance in examining lubricating compounds. Let us, however, remember that some resins yield insoluble compounds with sulphur chloride.

It is advisable to perform the experiments in duplicate, so as to obtain a check on the result; the difference should not exceed what we allow on an ordinary commercial analysis.

The washing with disulphide is carried under pressure; a foot blower is convenient, but by closing the top of the filter tube, the claspings it with the warm hand will be sufficient. The exhaust will in some cases give a further yield of solid products; in these cases, if a larger quantity of chloride be used in the first place, a harder product will be obtained. This is not to be recommended, unless for special purposes, because uniformity is aimed at in the result, and it is not desirable to alter the oils too much.

The exhaust is weighed after removal of disulphide, and when the weighings are constant this is deducted from the contents of the dish, by which we obtain the weight of insoluble solid products. This procedure is more simple and reliable than weighing the insoluble solid product. The smell and color of the exhaust will in many cases reveal what the oil itself is, in spite of blending, refining, etc.

The color and tenacity of the solid product is so very characteristic in most cases that no difficulty will be felt in deciding what the oil or mixture is; thus arachis oil in lard or olive oil can be instantly detected from cottonseed oil. Arachis oil is largely adulterated with cotton oil, and I have no doubt that in many cases where cotton is supposed to be present as an adulterant the intention of a manufacturer has been to use arachis oil. I propose to examine this double adulteration shortly.

Sulphur chloride is sometimes decomposed when added to an oil; the deposited sulphur is removed from the exhaust by washing with ether saturated with sulphur. The oily portion is taken up, leaving the sulphur; we then obtain the weight of the exhaust minus the sulphur. If much sulphur is present the exhaust has a cloudy white appearance. This indicates, generally, that the chloride is in excess.

* Chem. News, March 23, 1888, p. 113.

This method evidently was suggested by the article in Watt's old dictionary (Linseed oil), in which the action of sulphur chloride on flax oil is described (incorrectly, as Mr. Warren has shown).

The method was tried by Dr. C. A. Crampton during the lard investigations on a few of the samples submitted, viz :

- 5645. Cotton oil.
- 5624. Olive oil.
- 5620. Peanut oil.
- 5556. Fairbank's lard.
- 5626. Tallow stearine.
- 5591. Squire's lard.
- 5672. Hog's-head lard.

On adding the re-agent, mixing thoroughly, and heating on the water-bath the oils became perfectly solid. The lards did not become solid and the stearine was not affected at all.

5591. A pure standard lard was scarcely attacked by the re-agent.

5672. Rendered in laboratory from the head fat was more readily affected by the re-agent.

Mr. Crampton made a preliminary examination of the solid products formed, but came to the conclusion that they contained no sulphur. If it be true that only oleine is attacked by the chloride of sulphur and not palmitine nor stearine, then pure lard ought to give a partial product insoluble in ether and carbon disulphide. Yet lard so treated is practically soluble in the re-agents named. The vegetable oils appear to be easily attacked by the chloride of sulphur and the action of the re-agent does not seem to wholly depend on the amount of oleine present.

I think Mr. Warren's method may prove of great value qualitatively and perhaps quantitatively.

QUANTITATIVE DETERMINATION OF ADULTERANTS IN LARD.

Many attempts have been made to determine quantitatively the amount of adulterants in lard. These attempts have not been attended with much success. They may be classified as follows :

(1) By weight of undissolved residue when the mixed fat is treated with ether.

(2) By the relative intensity of color produced by sulphuric acid and other re-agents.

(3) By the relative quantities of silver or gold reduced, or intensity of coloration in Bechi's, Milliau's, and Hirschsohn's processes.

(4) By calculation from specific gravity.

(5) By calculation from iodine absorption.

(6) By calculation from refractive index.

(7) By determination of the insoluble matter produced by treatment with chloride of sulphur.

(8) By rise of temperature with sulphuric acid.

(9) By calculation from melting point.

(10) By the coloration produced by heating with nitric acid and albumen (Brullé's method).

In the peculiar conditions attending the analyses of mixed lards it is unnecessary to say that the most misleading results may be obtained by relying on any one of the above methods, and even when all are applied the real quantity of added adulterants may not be determined.

The processes indicated in Nos. 1, 2, and 3 of the foregoing classification may be dismissed without further discussion. They are entirely unreliable for any quantitative purpose.

(4) BY CALCULATION FROM SPECIFIC GRAVITY.

In the case of No. 4, approximate results could be reached were only one kind of adulterant used, the specific gravity of which, as in the case of cotton oil, is distinctly different from that of lard.

But if one adulterant be used like an oleo or lard stearine having a lower specific gravity, and another like cotton oil with a high one, the neutralizing effect of the two will render the results of the analysis unreliable.

Cotton oil, however, has a specific gravity considerably higher than that of a stearine is below the number for pure lard; hence a mixed lard containing equal portions of cotton oil and a stearine will have a higher specific gravity than pure lard. In point of fact, it may be said that where one of these adulterants is present in any notable quantity, say 15 to 30 per cent., the other is also present in proportions approximately known. It might be possible, therefore, to construct an arbitrary formula by which the disturbing effect of the second element could be allowed for. In this way some approximate number might be reached of the respective amounts of adulterants present.

Example :

Let specific gravity of pure lard	at 35° = .905
Let specific gravity of pure stearine	at 35° = .903
Let specific gravity of pure cotton oil	at 35° = .913

The theoretical specific gravity of a mixed lard composed of these bodies in the proportions stated would be as follows :

20 per cent. stearine	= .903 × 20 = 18.060
30 per cent. cotton oil	= .913 × 30 = 27.390
50 per cent. lard	= .905 × 50 = 45.250
<hr/> 100 per cent.	<hr/> = 90.700

Then theoretical specific gravity = .907.

It is usual to mix cotton oil and stearine in compound lards in the respective proportions mentioned above, viz, 1.5 parts to 1.*

* Testimony of George H. Webster, Report of Hearings before House Committee on Agriculture, p. 26.

The specific gravity of the mixture is therefore—

$$\text{Cotton oil, 1.5 parts} = .913 \times 1.5 = 1.3695$$

$$\text{Stearine, 1.0 part} = .903 \times 1.0 = .9030$$

$$\text{Mixture, 2.5 parts} = \underline{\underline{2.2725}}$$

Theoretical specific gravity = .909.

The following table, therefore, will give the approximate percentage of adulterations corresponding to the specific gravities noted :

Table showing approximate percentage of adulteration corresponding to different specific gravities when the adulterants are cotton oil and stearine in respective proportions of 1.5 to 1.

Observed specific gravity, at 35°.	Pure lard.	Adulterant.	Of which there is—	
			Cotton oil.	Stearine.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
.9050	100.00	0.00	0.00	0.00
.9055	87.50	12.50	7.50	5.00
.9060	75.00	25.00	15.00	10.00
.9065	62.50	37.50	22.50	15.00
.9070	50.00	50.00	30.00	20.00
.9075	37.50	62.50	37.50	25.00
.9080	25.00	75.00	45.00	30.00
.9085	12.50	87.50	52.50	35.00
.9090	0.00	100.00	60.00	40.00

A general expression for the calculations above when applied to other standards of temperature and actual results obtained may be easily devised. The general formula however will still rest on the assumption that the cotton oil and stearine are mixed in the proportions noted, and this will be found to be practically the case.

Let s = the observed specific gravity at t° .

a = specific gravity of pure lard at t° .

b = specific gravity of pure cotton oil at t° .

c = specific gravity of pure stearine at t° .

$$\frac{1.5b + c}{2.5} = \text{specific gravity of the mixed adulterants at } t^\circ.$$

x = per cent. of adulteration.

$$\text{Then } x = \frac{100(s-a)}{\frac{1.5b+c}{2.5} - a}$$

For illustration we may apply this formula to the data collected in tables which follow. Each analyst should carefully determine for himself, in a great number of samples, the true specific gravities of various

substances entering into the mixture at the temperature used by him as a standard.

Example.

Mean specific gravity of pure lard at 35°9053= <i>a</i>
Mean specific gravity of cotton oil at 35°9042= <i>b</i>
Mean specific gravity of stearine9015= <i>c</i>
Mean specific gravity of the cotton oil and stearine adulterant (calculated).	.9091 = $\frac{1.5\ b + c}{2.5}$

The mean specific gravity *s* of Armour's was .906.

Then

$$x = \frac{100 (.906 - .9053)}{.9091 - .9053}$$

Whence

$$x = 18.42 \text{ per cent.}$$

The mean specific gravity of Fairbank's lards was .9095. This shows a theoretical adulteration of over 100 per cent., or in other words a lard composed wholly of stearines and cotton oil, in which the oil is in slightly greater proportions than those indicated above. The iodine number obtained shows that the lard approximates such a composition.

(5) BY CALCULATION FROM IODINE ABSORPTION.

The determination of the percentage of iodine absorbed by a mixed lard taken alone can not lead to any just idea of the amount of adulterant added.

In the case of specific gravities the numbers for oleo-stearine and lard stearine are near together, viz, for 35° .900 and .902, respectively. But for iodine numbers the difference is very great. In the three samples of oleo-stearine examined the mean iodine number is 20.73 per cent. In the two samples of lard stearine analyzed it is 47.02 per cent. The mean number for cotton oils is 109.02 per cent., for lard, 62.48 per cent., and for prime steam lard, 62.86 per cent. In a mixture we may find all of these ingredients, and therefore the iodine number of such a mixture may approximate that of a pure lard.

When the iodine number of a supposed adulterated lard goes above 65 per cent. there are grave reasons for suspecting an adulteration with cotton oil, but a pure lard made from certain parts of the hog may show even a higher number.

If the microscopic examination show the presence of oleo-stearine, and cotton oil be revealed by the silver or gold tests, the complexity of the problem is less confusing. The iodine number may then reveal the approximate quantities of the two adulterants present.

For example:

1.5 parts of cotton oil at 109=	163.5
1. part of oleo-stearine at 20=	20.0
	<hr/>
2.5 parts	=183.5
1. part	= 73.4 per cent.

Now, a mixed lard whose iodine equivalent is about 64 per cent. (Armour's) can not be made of any considerable quantity of the above mixture and pure lard. It must contain a notable quantity of lard stearine.

For example :

40 parts of cotton oil and oleo-stearine at 74	=2960
30 parts of lard at	62=1860
30 parts of lard stearine at	47=1410
<hr/> 100 parts	<hr/> =6230

The theoretical iodine number of such a compound lard is therefore 62.30 per cent. The above hypothetical example, in the light of the analyses made, shows approximately the composition of a compound lard whose iodine number is not above 63 per cent.

In the Fairbank samples the mean iodine number is 85.31 per cent. The microscope revealed also the presence of oleo-stearine in these samples. They were presumably composed of cotton oil, lard, and oleo-stearines, and perhaps some lard. As was shown by the specific gravity they contained an excess of cotton oil. These mixtures may be represented by the following proportions:

10 parts oleo-stearine at 20	= 200
25 parts lard stearine at 47	=1175
65 parts cotton oil at 109	=7085
<hr/> 100 parts	<hr/> =8460
Theoretical iodine number = 84.60 per cent.	

No formulæ can be given for computing the proportions of ingredients from the quantity of iodine absorbed, except in the tentative way indicated above, but the value of the iodine number, when thus studied with other quantitative data, is sufficiently illustrated.

(6) *By calculation from the refractive index.*—Some valuable information concerning the quantitative composition of a mixed lard may be derived from a study of the refractive index.

The mean refractive index at 25° of the samples of lard examined is 1.4620; water at the same temperature showing 1.3300; for cotton oil the number is 1.4674; for oleo stearine, 1.4582; for lard stearine, 1.4594.

The determination of a much larger number of samples of the stearines would be desirable before deciding on a permanent standard, but the above numbers will serve provisionally.

	Points.
Difference between lard and cotton oil.....	+54
Difference between lard and oleo stearine	—38
Difference between lard and lard stearine.....	—24

It thus appears that the addition of cotton oil to a lard would raise its refractive index, while the addition of the stearines would lower it.

In general it appears that two parts of stearine would neutralize the effect of one part of cotton oil. A mixture of 1.5 parts of cotton oil and 1 part of mixed stearines would have the following theroretical index:

1.5 parts cotton oil at 1.4674	=2.2011
1 part stearines at 1.4588	=1.4588
—	—
2.5 parts	=3.6599
1 part	=1.4640

For a lard adulterated with the above-mixed adulterant we may use the following formulæ:

Let r = observed index at 25°

a = index at 25° of lard.

b = index at 25° of cotton oil.

c = index at 25° of stearine.

$\frac{1.5b+c}{2.5}$ = index at 25° of the mixed cotton oil and stearine.

x = per cent. of adulteration.

Then

$$x = \frac{100 (r-a)}{\frac{1.5b+c}{2.5} - a}.$$

As an illustration of this formula take the mean numbers obtained in the tables of samples for lard, cotton oil, stearines, and Armour's mixtures:

Mean index of Armour's samples	$r=1.4634$
Mean index of pure lards	$a=1.4620$
Mean index of cotton oils	$b=1.4674$
Mean index of stearines	$c=1.4588$

$$\text{Value of } \frac{1.5b+c}{2.5} = 1.4640$$

Then $x = .14 \div .0020 = 70$ per cent.

According to this formula Armour's samples would have only 30 per cent. of pure lard, a result which is contradicted by other data. I am inclined to believe that the examination of a larger number of samples of stearine may show a higher index and thus bring the results obtained by the application of the above formula more into harmony with the other data.

The index for the Fairbank samples, 1.4651, shows that in these mixtures cotton oil has been used in greater proportions than indicated above, thus corroborating the results obtained by the other methods of analysis. Judged by the index of refraction alone, on the assumption that this index for the stearines is not much different from that of lard,

the composition of a mixed lard is probably as truly indicated as by any other single method.

(7) *By determination of product formed by chloride of sulphur.*—Warren, in the articles already cited, has obtained some interesting results, and our own work has shown that much may be expected of a careful study of this process. Lack of time has prevented a full investigation and this will be made subsequently.

(8) *Rise of temperature with sulphuric acid.*—Valuable information relating to the composition of a mixed lard may be obtained by a study of rise of temperature of a given volume thereof when mixed with a definite quantity of strong sulphuric acid. The data obtained in our analyses are as follows:

Rise of temperature with—	Degrees.
Lard.....	41.5
Cotton oil.....	85.4
Oleo stearine.....	20.8
Lard stearine.....	37.7
Mean rise of temperature with the stearines.....	29.3

When the microscope reveals oleo stearine we may take the last number to represent the mean increment of temperature. For an adulterant composed of 1.5 parts of cotton oil and 1 part of stearine the mean rise of temperature would be 63°.

The apparent composition of a mixed lard on the above character of the adulterant would be illustrated by the following formula:

Let t = observed rise of temperature for sample.

a = rise of temperature for lard.

b = rise of temperature for cotton oil.

c = rise of temperature for stearine.

$\frac{1.5b+c}{2.5}$ = theoretical rise of temperature for the adulterant.

x = percentage of adulteration.

Then

$$x = \frac{100(t-a)}{\frac{1.5b+c}{2.5} - a}$$

This formula applied to the mean rise of temperature observed in Armour's samples gives the following result:

$$x = 23.3 \text{ per cent.}$$

Applied to Fairbank's samples it shows an adulteration of 76.3 per cent.

9. *Calculation from the melting point.*—The melting point of a fat is often of great value in helping to a correct understanding of its composition, but little reliance can be placed on it for quantitative purposes.

The different glycerides when mixed do not have a melting point which corresponds to the one theoretically calculated. For this reason equal mixtures of cotton seed oil and lard, instead of having a melting point of about 20°, really melt only at a much higher temperature. While, therefore, the determination of the melting point of a compound lard should not be omitted, it does not afford a basis for any reliable estimation of a quantitative nature.

10. *Heating with nitric acid and albumen.*—The coloration produced by heating the fat or oil under examination with nitric acid and albumen has also been proposed as a quantitative test. Although I have not tried this method quantitatively, I am of the opinion that it will be found of no greater value than the other color reactions already noted.

The Brullé test appears to be unaffected by free acid or rancidity, in which point it possesses an advantage over chloride of gold and in some cases over nitrate of silver.

SUMMARY.

From the methods already worked out as applied to the two classes of mixed lards examined the following general results are deducible, viz :

Method of examination by—	Sample from—	Per cent. of adulteration.
Specific gravity.....	Armour & Co.....	18.42
Refractive index.....do.....	70.00
Rise of temperature.....do.....	23.30
Specific gravity.....	Fairbank & Co.....	100.00
Refractive index.....do.....	100.00
Rise of temperature.....do.....	76.30

The mean percentage of adulteration for the Armour samples is 37.24. For the Fairbank samples it is 92.10.

It is not unusual to omit the percentage of lard stearine used in accounts given by manufacturers of the extent of adulteration. If we allow that one-third of the total adulterant is lard stearine the percentages of foreign fats in the Armour and Fairbank lards are 24.83 and 61.40 respectively.

In the foregoing discussion it has been assumed that the mean properties of a mixture of various glycerides are proportional to the quantities of each present. In the case of the melting point, we know that this is not the case, and the consideration of the melting point therefore as a factor in quantitative determinations has been omitted. It may be true that other properties are also unequally developed in a mixture, and this would add still another complication to the problem.

In the present state of our knowledge the chemist is unable to express definitely the degree of adulteration which a sample of lard has suffered. He can state with confidence whether or not a given sample is adul-

terated, and in the comparison of two widely different sets of samples—such as were obtained from Armour & Co. and Fairbank & Co.—he may safely say that one is adulterated to a greater degree than the other. Further than this the present state of our knowledge will not permit us to go.

RESULTS OF ANALYSES.

The samples of lards and lard compounds, whose analyses follow, were furnished by different persons, each sample usually accompanied by an affidavit showing where it was bought, name of sample, etc., or were purchased in open market by agents of the chemical division or rendered in the laboratory.

The classification was made as follows :

- (1) Lards known or believed to be pure hog grease.
- (2) Prime steam lards from Chicago Board of Trade.
- (3) Lards of miscellaneous origin, both pure and adulterated.
- (4) Cotton oils from different localities.
- (5) Crude cotton oils and foots.
- (6) Oleo, lard, and cotton-oil stearines.
- (7) Mixed lards from Armour & Co., Chicago, Ill.
- (8) Mixed lards from N. K. Fairbank & Co., Chicago, Ill.
- (9) Miscellaneous oils.

Each sample is indicated by a number, and with each table is a list of these numbers, with a full description of the name of the sample, and place or person from whom obtained.

In the foregoing pages the analytical data obtained have been rather fully discussed, and only such explanatory items will be added here as may help to elucidate the tables. The amount of analytical work which has been done, as will be seen, is very large. While absolute accuracy has not been obtained, it is believed the data in general may be accepted as reliable.

Such an amount of work can only be accomplished by the united labors of several participators, and this, of course, magnifies the personal error to a certain degree.

SAMPLE OF AFFIDAVITS.

Many of the samples were accompanied by affidavits, which it is not necessary to reproduce in full. Their general tenor may be seen from the four following :

5646.

STATE OF NEW YORK,
City and County of New York, ss :

Carl Dreier, being duly sworn, says: That he is a manager with N. K. Fairbank & Co., of Chicago; that it is the custom of the said firm to keep a stock of their refined

lard on hand at their manufactory in Chicago for sale to the domestic and foreign trade; that the accompanying package of refined lard in the original can marked "H" was taken from the stock of the said firm on hand at the said manufactory, and is the same as that sold regularly by the said firm in the domestic and foreign markets as refined lard; that the said package was taken at random from the said stock without special selection, and that all of said stock is alike as to composition and quality as that usually and regularly sold in the domestic and foreign markets by the said firm and known as refined lard; that the said package was immediately fastened up, sealed, and marked "H" by this deponent, and that no ingredient or thing has been added to or extracted from the same; and deponent further says that the said package is a true, genuine, and fair sample of the refined lard as regularly made and sold by the said firm, and that it was not specially prepared for testing, analysis, or exhibition purposes, and that it is in every sense a genuine sample of refined lard.

CARL DREIER.

Sworn to before me this 16th day of February, 1888.

ALFRED JUNTZTH,
Notary Public, New York County.

[Affidavit to accompany Nos. 5550, 5551, and 5552.]


I, Walter L. Hill, on oath affirm and declare that I purchased on the seventh day of February, A. D. 1888, in the city of Washington, D. C., in the open market, the following packages of pail lard and paid for the same the price set against the respective items; that the names of the parties in whose places of business the same were purchased were as follows, to wit:

One (1) three (3) pound pail of lard, marked "Jacob Schaefer, & Co., Baltimore. Pure natural lard, bought of Henry W. Kem & Co., No. 12 Centre Market, Washington, D. C., price thirty-five (35) cents."

One (1) three (3) pound pail of lard, marked "Chas. G. Kriel, cream leaf lard, Baltimore, price thirty (30) cents;" bought of Jas. Schneider, No. 529 Centre Market, Washington, D. C.

One (1) three (3) pound pail of lard, marked "Armour & Co., pure refined family lard, Chicago," bought of E. C. Ford & Son, No. 609 Centre Market, Washington, D. C.; price thirty-five (35) cents.

And I, the said Hill, further on oath affirm and declare, that I likewise purchased on the eighth day of February, A. D. 1888, in the said city of Washington, in the open market, the following package of pail lard and paid for the same the price set against the said item, and that the name of the party in whose place of business the same was purchased is as follows, to wit:

One (1) five (5) pound pail of lard, marked "G. Cassard & Son, best refined lard, Baltimore," with a star, viz.,  bought of Hume, Cleary & Co., No. 807 Pennsylvania avenue, Washington, D. C.; price fifty-five (55) cents.

That I took said samples and delivered them to Professor S. P. Sharpless in the original packages, as purchased, and that the same were not in way tampered with by me.

W. L. HILL.

CITY OF WASHINGTON,
District of Columbia, ss.

Subscribed and sworn to before me this the 10th day of February, A. D. 1888.

ROBERT R. SHELLABARGER,
Notary Public, D. C.

[Affidavit to accompany Nos. 5662-66.]

I hereby certify that certain samples of lard, numbered two to six inclusive, and marked "From C. H. S. Mixer, Chicago, 2-8-88," were prime steam lard, and of the quality known as standard lard by the Board of Trade of the city of Chicago. Said samples were from five different lots of lard, and were made by as many different packers, and the samples fairly represented the different lots from which they were taken. The said five samples of lard were drawn on the eighteenth day of February last, and were on the same day shipped per express and were addressed to "Prof. Sharpless, Riggs House, Washington, D. C."

C. H. S. MIXER,
Chief Inspector of Provisions, Chicago Board of Trade.

Personally appeared C. H. S. Mixer, signer of the foregoing statement, who made solemn oath to the truth of the same, this fifth day of March, 1888.

R. S. WORTHINGTON,
Notary Public.

[Affidavit to accompany No. 5610.]

STATE OF ILLINOIS,

Cook County, ss:

Geo. H. Webster, being first duly sworn, on oath deposes and says, that he is a member of the firm of Armour & Co., doing business in the city of Chicago, Cook County, Illinois, and that he makes this affidavit on behalf of himself and his co-partners in said firm; that he has seen and knows the five pound tin of lard which is herewith submitted to Dr. H. W. Wiley, chemist of the Department of Agriculture of the United States, for analysis, and that the same was manufactured by the said firm of Armour & Co., in accordance with their regular formula for the manufacture of refined lard for foreign trade.

GEO. H. WEBSTER.

Sworn and subscribed to before me this 11th day of February, 1888.

CHARLES F. LANGDON,
Notary Public in and for Cook Co., Ills.

TABLE NO. 17.—*Pure lards.*

No.	Specific gravity at 35°.	Melting point Co.	Melting point of fat acids, Co.	Crystallizing temperature of fat acids, Co.	Color reactions with—		Refractive index at 25°.	Rise of temperature with H_2SO_4 Co.	Reaction with silver nitrate.		Water.	Microscopic indications.	Iodine absorbed.
					H_2SO_4 .	HNO_3 .			Bechi.	Millian.			
5550	.9054	39.6	42.0	39.0	1.4617	42.1	Light red.	Trace of reduction.	P. ct.	Lard stearine.	P. ct.
5565	.9055	39.6	42.6	39.8	1.4630	39.8	do	do	.045	do	58.35
5566	.9057	41.6	43.0	40.4	1.4605	39.7	Trace of color	do	.165	do	59.60
5591	.9038	41.2	45.0	41.2	Trace of pink.	Light color	1.4625	41.1	do	do	.090	do	58.70
5592	.9043	43.6	41.3	Faint pink.	Trace	1.4622	41.4	do	do	Lard stearine.	58.43
5593	.9045	45.1	46.6	41.5	do	do	1.4626	40.9	do	do	do	56.93
5600	Trace of color	No color	1.4633	Faint brown	do	Lard, possibly tallow stearine.	60.94
5601	.9071	41.5	40.0	do	do	do	Lard stearine.	61.28
5606	.9067	39.8	41.4	38.9	Faint brown pink.	Light pink	44.9	Light brown	Trace of reduction.	Lard, possibly tallow stearine.	60.02
5608	.9051	40.4	43.6	40.0	Light brown pink.	Faint color	1.4610	Light red brown.	No reduction	Lard stearine.	69.79
5655	.9028	44.9	42.8	40.4	Light yellow.	37.1	Faint red	Trace of reduction.	.025	do	53.04
5656	.9042	38.7	46.2	42.7	Marked red brown.	Slight coloration.	1.4621	42.0	Light brown	do	.095	do	60.09
5657	.9037	43.9	42.2	39.7	Trace of yellow.	do	40.8	No color	do	.050	do	55.85
5658	.9056	39.0	41.4	36.6	Trace	No color.	42.6	do	do	.075	do	64.45
5672	.9066	35.5	40.0	36.4	1.4623	46.5	Faint color.	do	do	85.03
5673	.9045	44.0	46.4	42.7	1.4606	36.6	Marked red.	do	do	57.34
5674	.9052	42.5	44.0	41.3	1.4617	43.7	Slight color.	do	do	62.55
5676	.9088	35.1	36.9	32.1	1.4639	No color	do	do	77.28
5679	.9067	36.0	48.4	38.0	1.4618	42.7	Slight color.	Jelly	64.98
Means	.9053	40.7	43.3	39.6	1.4620	41.5077	62.48

NOTES ON TABLE NO. 17.*

This table includes the analyses of nineteen samples of lard, which both in pedigree and properties appear to be pure hog grease, taken from those parts of the animal usually devoted to lard-making.

Under the head of miscellaneous lards, there are other samples which appear to be pure lard, but the evidence was not in all cases sufficiently conclusive to warrant their incorporation in this table.

Number and description of samples in Table No. 17.

Number.

5550. "Pure Natural Lard," brand of Jacob C. Shafer & Co.; purchased in Washington. Affidavit of Walter L. Hill.
5565. "Best Refined Lard," brand of G. Cassard & Son, Baltimore, Md.; purchased in Savannah, Ga. Affidavit of Isaac G. Haas.
5566. "Leaf Lard," brand of Rohe & Bro., New York; purchased in Savannah, Ga. Affidavit of Isaac G. Haas.
5591. "Pure Leaf Lard," brand of John P. Squire & Co., Boston, Mass.; from manufacturer.
5592. Same as above.
5593. Same as above.
5600. "Pure Unadulterated Lard," brand of F. Whittaker & Sons, Saint Louis, Mo. Affidavit of R. A. Hamilton.
5601. "Pure Unadulterated Honest Refined Lard," brand and affidavit same as above.
5606. "Best Refined Lard," brand of G. Cassard & Sons; purchased in Washington. Affidavit of Walter L. Hill.
5607. "Pure Country Lard," rendered by L. Entriken, West Chester, Pa.
5655. "Pure Leaf Lard," from Deerfoot Farm Company, Boston, Mass. Affidavit of Frank W. Bennett.
5656. "Choice Leaf Lard," brand of Charles H. North & Co., Boston, Mass.; purchased of manufacturer. Affidavit of Frank W. Bennett.
5656. No brand, leaf lard, of Niles Brothers, Boston, Mass. Affidavit of Frank W. Bennett.
5657. "Pure Leaf Lard," brand of Sperry & Barnes, New Haven, Conn.; purchased in Boston, Mass.
5672. Lard from head of hog, rendered in United States Department of Agriculture.
5673. Intestine lard rendered in laboratory.
5674. Leaf lard, rendered in United States Department of Agriculture.
5676. Lard from pigs' feet, from David Wesson, Chicago, Ill.
5679. "Pure Natural Lard," brand of Jacob C. Shafer & Co.; purchased from manufacturer.

In specific gravity 5655 resembles lard stearine. Pigs' feet lard, 5676, should be considered apart, since no lard of commerce is ever made exclusively of pigs' feet. It represents the other extreme of specific gravity.

The highest melting point is shown by 5593 and the lowest by 5676.

The highest color with acids was shown by 5656 and with silver nitrate by 5673.

Judged by density alone, 5655 and 5676 would have pronounced adulterated, the former with stearine, the latter with cotton oil. The latter sample would also be looked on as suspected by reason of its high re-

* In serial order from page 120, part first.

fractive index. Both 5676 and 5672 show iodine numbers which would lead the analyst to look for a high percentage of cotton oil.

Leaving out these samples made from special parts of the animal, the mean iodine number for the other samples would be materially reduced.

It appears that the true number for lards of commerce would be about 60.

In addition to its low specific gravity No. 5655 is abnormal, both in the slight rise of temperature it gives with sulphuric acid and its low iodine number. In all three properties, viz, specific gravity, rise of temperature with sulphuric acid, and iodine number, it indicates the presence of lard stearine, or that it is made from some special part of the fat.

Prime steam lard. Table No. 18.

No.

5629. Prime steam lard purchased in Saint Louis, Mo. Affidavit of R. A. Hamilton.

5639. Prime lard, steam-rendered, from D. E. Fox, taken by Chicago inspector. Affidavit of Carl Dreier.

5640. Prime steam lard from D. E. Fox, taken by Chicago inspector. Affidavit of Carl Dreier.

5641. Prime steam lard from D. E. Fox, taken by Chicago inspector. Affidavit of Carl Dreier.

5642. Prime steam lard from D. E. Fox, taken by Chicago inspector. Affidavit of Carl Dreier.

5650. Prime steam lard from John P. Squire & Co., Boston, Mass.

5662. Prime steam lard taken by Chicago inspector. Affidavit of C. H. S. Mixer.

5663. Same as above.

5664. Same as above.

5665. Same as above.

5666. Same as above.

TABLE NO. 18.—*Steam lards.*

No.	Specific gravity at 35° C.	Melting point, C°.	Melting point of fat acids, C°.	Crystallizing temperature of fat acids, C°.	Color reactions with—		Refractive index at 25° C.	Rise of temperature with H ₂ SO ₄ , C°.	Reaction with silver nitrate—		Water.	Microscopic indication.	Iodine absorbed.
					H ₂ SO ₄ .	HNO ₃ .			Beech.	Millau.			
5629	.9052	38.4	41.8	39.53	Slight color.....	Light yellow.....	1.4616	33.7	279.06	Faint brown.....	Trace of reduction.	Lard stearine...	<i>Per et.</i> 63.84
5639	.9051	39.0	41.8	38.95	Very light color.....	Light red.....	1.4612	40.9	286.84	Light brown.....	do.....	do.....	62.14
5640	.9054	40.5	42.6	39.65	Light red.....	Marked pink.....	1.4617	39.6	276.14	do.....	do.....	do.....	62.33
5641	.9051	42.9	43.0	35.40	Faint color.....	Bright pink.....	1.4608	38.8	288.87	Faint brown.....	Jelly.....	do.....	66.47
5642	.9069	32.3	42.2	39.00	Trace.....	Marked pink.....	1.4622	42.1	282.42	Slight discoloration.	Trace of reduction.	do.....	62.35
5650	.9046	37.7	41.4	38.49	Light red-brown.....	Pale yellow-brown.....	1.4616	41.1	277.20	Slight color.....	Jelly.....	do.....	62.22
5662	.9046	38.3	42.0	39.30	Trace.....	Faint pink.....	1.4675	41.6	286.87	do.....	Trace of reduction.	do.....	61.95
5663	.9065	29.8	42.2	38.05	do.....	do.....	1.4641	41.3	290.05	do.....	do.....	do.....	63.82
5664	.9050	31.5	42.0	38.05	do.....	Trace.....	1.4611	39.3	290.05	do.....	do.....	do.....	60.34
5665	.9067	42.0	do.....	do.....	1.4616	41.7	do.....	Trace of reduction.	Lard stearine...	65.12
5666	.9056	38.9	42.4	39.10	do.....	do.....	1.4614	39.5	283.61	do.....	do.....	do.....	60.85
	.9053	37.0	42.1	38.60	1.4623	39.9	283.45	62.86

NOTES ON TABLE 18.

As can be seen by the description of the samples, Table No. 18 contains analyses of fairly good specimens of the prime steam lard of the Chicago market.

The specific gravities of the samples are very near together, differing in any case at most only .0014 from the mean.

The variations in the melting point are more marked, and in Nos. 5663 and 5664 we notice results which are quite anomalous. In No. 5663 the melting point and crystallizing point of the fat acids are comparable with the mean results, which leads to the suspicion of some inadvertent error in determining the melting point of the glycerides. The mean refractive index is slightly higher than that for lards made in other ways. The iodine number is also higher than for pure lards of different origin, especially with the exceptions noted in table No. 18.

When the rise of temperature with sulphuric acid, however, is considered, a lower number is obtained than in No. 17. The numbers for single samples show a close agreement with the exception of 5629, one of the two samples in the table not obtained in Chicago.

As a general observation it may be stated that the steam lards of commerce have a more constant composition than pure lards made in other ways and from more restricted portions of the animal.

Steam lards have a distinctively strong odor which distinguishes them from lards rendered in open kettles at low temperatures and from selected portions of the fat.

Cottonseed oil.

No.

5553. Cotton oil, from F. Whittaker & Sons, Saint Louis, Mo.

5554. Yellow cotton oil, from Naphey & Co., Philadelphia, Pa.

5555. White or refined cotton oil, same source as above.

5615. Summer yellow, received from D. E. Fox.

5616. Summer white, received from D. E. Fox.

5618. From Z. D. Gilman, Washington, D. C., marked Olive Oil Sublime.

5619. Cotton oil, same source as above.

5625. Cotton oil, purchased in Boston, Mass. Affidavit of Walter L. Hill.

5628. Cotton oil, purchased in Saint Louis, Mo. Affidavit of D. H. Kennett.

5645. Prime cotton oil, from N. K. Fairbank & Co., Chicago, Ill. Affidavit of Carl Dreier.

5647. Light yellow cotton oil, Maginnis Oil Works, New Orleans, La. Affidavit of Carson Mudge.

5648. Light yellow cotton oil, purchased from Union Oil Company, New Orleans, La. Affidavit of Carson Mudge.

5649. Light yellow cotton oil, purchased from Delta Oil Works, New Orleans, La. Affidavit of Carson Mudge.

5661. Cotton oil, taken from car by C. H. S. Mixer, in Chicago, Ill. Affidavit of C. H. S. Mixer.

5683. Summer yellow cotton oil, from Southern Cotton Oil Trust.

5684. Summer white cotton oil, Southern Cotton Oil Trust.

5685. Winter yellow cotton oil, Southern Cotton Oil Trust.

5686. Winter white cotton oil, Southern Cotton Oil Trust.

TABLE No. 19.—*Cotton oils.*

No.	Specific gravity at 35°.	Melting point of fat acids C°.	Crystallizing temperature fat acids, C°.	Color reactions with—		Refractive index at 25°.	Rise of temperature with H ₂ SO ₄ , C°.	Saponification equivalent.	Reaction with silver nitrate.		Water.	Iodine absorbed.
				H ₂ SO ₄ .	HNO ₃ .				Bechi.	Millian.		
5552	Black-red	Per cent.	Per cent.
5554	.9140	37.2	32.8	1.4671	81.6	Very black	Marked reduction	.125	113.90
5555	.9139	38.6	35.1	Green-brown	1.4680	82.1	do075	110.06
5615	.9134	39.4	34.5	Marked brown-red	Marked red-yellow.	1.4677	85.8	Black-brown-green tint.	Slight reduction	109.28
5616	.9149	39.6	35.1	Green-yellow-brown	do	1.4677	86.3	do	Heavy reduction	108.73
5618	.9143	34.6	32.4	Brown-red	Yellow-red	1.4677	88.4	do	No reduction	105.59
5619	.9149	36.6	32.4	Very dark red-brown	Dark yellow-red	1.4682	90.2	do	do	111.46
5655	.9153	36.6	32.3	Dark red-brown	Deep red	1.4683	89.8	Very black	113.39
5638	.9154	37.4	30.9	Marked red-brown	Red-brown	1.4681	83.3	Very black-brown-blue tint.	Trace	105.31
5645	.9140	42.6	Marked brown	Light red	1.4685	90.1	286.25	Very black-brown	Jelly	.050	116.97
5647	.9136	41.4	35.6	Red-brown	Red-brown	1.4670	80.4	278.79	Black	Slight reduction	.070	105.08
5648	.9137	35.2	do	do	1.4670	80.9	286.80	do055	105.42
5649	.9133	42.5	35.2	Light yellow	do	1.4664	81.9	282.05	do	Jelly	.050	100.94
5661	.9141	44.4	34.4	Marked yellow	1.4705	87.3	287.03	Dark brown	No reduction	.020	106.02
6683	.9134	38.8	34.6	1.4669	85.4	Deep red-brown	Heavy reduction	109.53
5684	.9132	84.4	286.10	do	Slight reduction	106.19
5685	.9154	36.8	30.5	1.4675	85.5	279.59	do	do	114.40
5686	.9138	35.8	31.8	1.4680	do	No reduction	111.06
Means.	.9142	38.8	33.5	1.4678	86.7	283.80064	109.06

NOTES ON TABLE NO. 19.

The cotton oils examined are believed to represent very accurately the oils used in the adulteration of lards. The samples were mostly taken from large reservoirs and hence better represent a mean value than if derived from small quantities of the material.

The specific gravity of the samples is remarkably uniform, the greatest variation from the mean being $+0.0011$.

The high melting and crystallizing points of the fat acids are remarkable characteristics when the low temperature at which cotton oil is a solid is taken into consideration. The figures show how independent these acids are of the glyceride in many of their physical properties.

The high refractive index of cotton oil has already been noted. In No. 5661 this index is far above the mean, while in No. 5649 it falls considerably below. With these exceptions there is a fair agreement among the indices of the remaining samples.

The great rise of temperature shown by cotton oil in contact with sulphuric acid is fully illustrated by the numbers in the table. These numbers are fairly concordant. The greatest departures from the mean are -6.3° and $+3.5^{\circ}$.

By the silver nitrate test the original samples were easily recognized as cotton oil, while with the same test applied to the free acids, the results, as already indicated, were not so decisive. The probable reason for this has already been mentioned.

In the samples marked "jelly" in all the tables the silver test would not work on account of a gelatinous precipitate, due doubtless to the formation of a salt in the samples, arising from the union of an organic acid with the silver. This organic acid was separated, but not in sufficient quantity to determine its properties. The high iodine number is another characteristic to be noted. Nos. 5649 and 5645 show the greatest departures from the mean.

Stearines.

No.

- 5612. Prime oleo-stearine, made and used by Armour & Co., Chicago, Ill. Affidavit of George H. Webster.
- 5613. Prime lard stearine, made and used by Armour & Co., Chicago, Ill. Affidavit of George H. Webster.
- 5626. Oleo-stearine from John Rearden & Sons, Boston, Mass. Affidavit of Walter L. Hill.
- 5630. Yellow cottonseed oil stearine, brand of N. K. Fairbank & Co., Chicago, Ill., from E. Richards.
- 5631. Cotton-seed stearine obtained by Z. D. Gilman, from E. Richards.
- 5643. Prime lard stearine, from N. K. Fairbank & Co., Chicago, Ill. Affidavit of Carl Dreier.
- 5644. Oleo-stearine, from N. K. Fairbank & Co., Chicago, Ill. Affidavit of Carl Dreier.
- 5652. Dead-hog stearine, from John P. Squire, Boston, Mass.
- 5675. Sample from David Wesson, supposed to be cottonseed oil stearine.
- 5680. Stearine from white cottonseed oil, from Southern Cotton Oil Trust.
- 5681. Stearine from yellow cottonseed oil, from Southern Cotton Oil Trust.

TABLE NO. 20.—*Stearines used in lard adulteration.*

No.	Specific gravity at 35°.	Melting point, C°.	Color reactions with—		Refractive index at 25°.	Rise of temperature with H ₂ SO ₄ C°.	Saponification equivalent.	Reaction with silver nitrate.		Water.	Microscopic indication.	Iodine absorbed.
			H ₂ SO ₄ .	HNO ₃ .				Bechi.	Millau.			
5612	.9006	51.9	None	Trace of yellow	No color	<i>Per et.</i>	Tallow stearine...	<i>Per et.</i>
5613	.9031	44.3	Trace	Trace	*1.4594	Faint blue580	Tallow stearine...	17.38
5626	.9011	Marked pink	Light yellow	1.4579	21.7	Faint pink050	Lard stearine...	44.24
5630	.9175	Dark brown	Quite red	Deep brown-red295	Tallow stearine...	26.81
5631	.9142	do	Red yellow	Very black brown	89.54
5643	.9030	45.8	None	Trace of pink	37.7	Faint color055	Lard stearine...	85.28
5644	.8998	53.8	Trace	Light yellow	*1.4584	20.0	No color330	Tallow stearine...	49.78
5652	.9091	38.2	Very dark brown	Very dark red	do	More trace.	.850	18.00
5675	.9149	1.4682	Deep red-brown	45.35
5680	1.4664	80.4	do	Light reduction.	92.78
5681	1.4659	64.0	203.11	Very black	Heavy reduction, very black.	99.39
												91.90

* See notes.

NOTES ON TABLE NO. 20.

The number of stearines examined is not large enough to fix a standard of comparison. Those of interest in the study of mixed lard are the prime lard and oleo-stearines. The cotton-oil stearines are not very extensively used in lard adulterations. The dead hog-grease stearines are never used for this purpose.

The specific gravity of the oleo-stearines is slightly below that of the prime lard stearine samples, and both are less than the specific gravity of pure lard. The melting point of both stearines is above that of lard. In the column containing the refractive indices those marked with an asterisk are of samples of different origin from the remainder of the table, but they are believed to be good representative samples. The mean index, however, should be determined from a much larger number of samples.* It will doubtless be found to be somewhat lower than the index of pure lards taken at the same temperature.

The rise of temperature on mixing with sulphuric acid is much less in the case of the stearines than with pure lard, showing that this phenomenon is chiefly characteristic of oleine. The stearines of cotton oil, however, show an increase of temperature comparable with that of the original oil, and lard stearine a much greater increase than oleo.

The low iodine equivalent of oleo-stearine has already been noticed and is strikingly shown by the data in the table. The cotton-oil stearines show a marked decrease from the numbers obtained for the oil itself.

Armour's lards.

No.

- 5552. Pure refined family lard, Washington, D. C. Affidavit of W. L. Hill.
- 5557. Kettle refined lard, Mobile, Ala. Affidavit of F. H. McLarney.
- 5559. Pure refined family lard, Macon, Ga. Affidavit of T. Skelton Jones.
- 5561. Choice refined family lard, Macon, Ga. Affidavit of T. Skelton Jones.
- 5562. Choice refined family lard, Kansas City, Mo. Affidavit of T. Skelton Jones.
- 5564. Pure refined family lard, Savannah, Ga. Affidavit of Isaac G. Haas.
- 5572. Choice family lard, Saint Louis, Mo.
- 5581. Choice family lard, Kansas City, Mo. Affidavit of E. K. Converse.
- 5584. Pure refined family lard, New Orleans, La. Affidavit of E. K. Converse.
- 5595. Pure refined family lard, Philadelphia, Pa. Affidavit of W. L. Hill.
- 5610. Pure refined family lard. Affidavit of George H. Webster.
- 5611. Pure refined family lard. Affidavit of George H. Webster.
- 5653. Superior compound lard, Boston, Mass. Affidavit of Frank W. Bennett.

* The analytical data show that oleine has a higher refractive index than stearine or palmitine.

TABLE NO. 21—*Armour's lards.*

No.	Specific grav. at 35°.	Melting point, C°.	Melting point of fat acids, C°.	Crystal- lizing tempera- ture of fat acids, C°.	Color reactions with—		Refrac- tive index, at 25°.	Rise of temper- ature with H ₂ SO ₄ , C°.	Saponi- fication with equiv- alent.	Reaction with silver nitrate.		Water.	Microscopic in- dications.	Iodine ab- sorbed.
					H ₂ SO ₄ .	HNO ₃				Eechi.	Millian.			
552....	.9064	39.7	38.5	1.4668	42.1	273.66	Almost black....	<i>Per ct.</i> .140	Lard and tallow stearine.	<i>Per ct.</i> 71.19
5557....	.9061	43.3	43.0	41.1	1.4626	44.3	273.52	Slight color....	Slight reduction	.025	Lard stearine....	61.18
5559....	.9037	39.5	42.6	39.9	1.4629	42.5	Quite brown....	Jelly.....	.060	do.....	62.08
5561....	41.9	1.4652	58.9	276.37	Very black....260	61.67
5562....	.9057	41.7	42.4	40.1	1.4617	42.1	273.44	Brown.....	Marked reduc- tion.	Tallow stearine.	61.67
5564....	.9063	38.9	43.0	39.3	1.4616	42.5	275.74	Quite brown....120	Lard stearine....	63.50
5572....	.9045	42.5	41.8	Dark brown....	Dirty brown	1.4646	49.9	272.84	Red-brown....	Marked reduc- tion.	.055	Tallow stearine.	54.11
5581....	.9063	40.7	43.4	39.3	Light red-brown	Red-brown....	1.4622	44.7	276.86	Black-green tint	Slight reduction	.020	Lard and tallow stearine.	63.97
5584....	.9066	39.5	42.6	39.4	Slight color....	Faint red....	1.4628	56.1	279.42	Brown-red....	do.....	.105	do.....	66.73
5595....	.9063	38.9	43.4	Dirty brown....	Yellow red	1.4618	44.0	Black-brown- green tint.	do.....	67.52
5610....	.9064	41.0	39.8	Marked brown....	do.....	1.4626	48.0	do.....	65.41
5611....	.9068	40.0	39.5	Light brown....	Light yel- low red.	1.4626	44.7	do.....	Trace of reduc- tion.	62.00
5653....	.9055	39.7	43.4	40.9	Quite brown....	Pink-red....	1.4618	44.5	Dark brown....	65.46
Means	.9060	40.6	42.8	39.8	1.4634	46.5	275.23098	63.58

NOTES ON TABLE 21.

The samples analyzed show that the compound lards manufactured by Armour & Co., of Chicago, have nearly a uniform constitution. The specific gravities of the various samples differ only slightly from the mean. The maximum difference is in No. 5572, viz, — .0015, and the maximum + difference in No. 5611, viz, .0008. The melting points are also remarkably constant, the two maximum variations of a positive and negative sign being + 2.7°, in No. 5557 and — 1.7° in Nos. 5564 and 5595. The same agreement is also noticed in the melting and crystallizing points of the fat acids. The refractive index in most cases is only slightly above that of pure lard, showing only a small addition of cotton oil, or a correction of the index thereof by a corresponding addition of a stearine with low index.

The rise of temperature with sulphuric acid shows only notable variations in Nos. 5561 and 5584.

In the latter of these the iodine number is correspondingly increased, but not so in the former. This is only another of the numerous illustrations of the analytical perplexities encountered in the study of mixed glycerides.

The reactions with silver nitrate reveal very well, in almost every instance, the presence of cotton oil, but in some cases the phenomena of reduction of silver are not sufficiently developed to distinguish the samples from lard containing enough of impurity other than cotton oil to give a color with silver nitrate. The silver test applied to the free acids did not afford satisfactory results; a fact which has already been noted, and its possible explanation given.

In the iodine numbers, those obtained for Nos. 5552, 5584, and 5595 are much above the mean, while in one case, No. 5572, the percentage of iodine absorbed, viz, of 54.11, would indicate the admixture of a larger quantity than usual of oleo-stearine.

Fairbank's lard.

No.

5553. Prime refined family lard, purchased in Mobile, Ala. Affidavit of F. H. McLarney.

5561. Prime refined family lard, purchased in Macon, Ga. Affidavit of T. Skelton Jones.

5563. Choice refined family lard, purchased in Savannah Ga. Affidavit of Isaac G. Haas.

5567. Prime refined family lard, purchased in Dallas, Tex. Affidavit of Thomas F. McEnnis.

5569. Prime refined family lard, purchased in Saint Louis, Mo.

5573. Prime refined family lard, purchased in Saint Louis, Mo.

5574. Prime refined family lard, purchased in Atlanta, Ga.

5576. Prime refined family lard, purchased in New Orleans, La. Affidavit of E. K. Converse.

5586. Prime refined leaf-lard, purchased in Norfolk, Va. Affidavit of W. B. Pearman.

5596. Prime refined family lard, purchased in Philadelphia, Pa. Affidavit of W. L. Hill.
5634. No brand; original small package "Y." Affidavit of William T. Wells.
5635. No brand; original large package "Z." Affidavit of William T. Wells.
5636. "X" prime refined family lard, purchased in New York. Affidavit of William T. Wells.
5637. "XX" prime refined family lard, purchased in New York. Affidavit of William T. Wells.
5638. "S" Cuba export refined lard, purchased in New York. Affidavit of William T. Wells.
5646. Prime refined family lard, from D. E. Fox. Affidavit of Carl Dreier.
5654. Compound lard purchased in Boston, Mass. Affidavit of Frank W. Bennett.

TABLE 22.—N. K. Fairbank's Lards.

No.	Specific gravity at 35°c.	Melting point, °C.	Melting point of fat acids, °C.	Crystallizing temperature of fat acids, °C.	Color reactions with—		Refractive index at 25°.	Rise of temperature with H_2SO_4 .	Saponification equivalent.		Reaction with silver nitrate.		Water.	Microscopic indication.	Iodine absorbed.
					H_2SO_4 .	HNO_3 .			Bechi.	Millan.					
5558....	.9113	36.4	40.2	36.7	1.4656	58.1	278.37	Very black	Heavy reduction, black.	<i>P. et.</i> .28	Tallow stearine.	<i>P. et.</i> 90.56
5561....	.9099	41.9	41.8	37.5	1.4652	58.9	276.37	do	Marked reduction.	.26	do	83.78
5563....	.9105	38.8	40.2	35.5	1.4700	58.2	278.93	Quite black24	do	92.62
5567....	.9108	38.1	1.4692	62.9	do31	do	93.34
5569....	.9123	34.4	35.7	Brown	Red brown	1.4653	61.5	274.57	Green-black	Slight reduction	.25	do	92.22
5573....	.9109	31.3	36.0	Light brown	Light red-brown	1.4663	54.2	do13	Lard and tallow stearine.	82.47
5574....	42.5	30.0	Very dark brown	Pronounced red	1.4656	51.3	277.20	do	Slight reduction	.03	Tallow stearine	70.47
5575....	.9109	35.1	40.0	Decided brown	Dirty brown	1.4659	68.8	276.26	Very black	Marked reduction.	.27	do	94.78
5583....	do	Deep brown-red	Green-black20	Lard stearine	88.92
5596....	.9094	39.3	35.4	Dirty brown	Yellow-red	1.4648	59.0	278.96	Black-brown	Tallow stearine.	88.17
5634....	.9087	39.5	42.4	39.5	Red-brown	Red	1.4633	56.5	Deep black-green tint.	Heavy reduction	do	80.89
5635....	.9052	39.0	41.6	37.7	do	Light red	1.4638	55.0	281.58	do	Marked reduction.	.09	do	78.24
5636....	.9086	35.5	39.4	34.7	Light red-brown	do	1.4646	57.9	281.69	do	Heavy reduction	do	89.93
5637....	.9098	35.5	40.8	37.0	do	Marked red	1.4648	59.2	280.50	do	Marked reduction.	do	87.22
5638....	.9075	43.2	42.6	39.5	Red-brown	Red	1.4631	52.9	280.16	Black-brown	do	75.57
5646....	.9075	40.4	42.2	38.3	Brown	Yellow-brown	1.4629	53.1	280.49	Dark brown-green tint.	Marked reduction.	76.24
5654....	.9088	38.9	45.4	42.9	Marked brown	Red-brown	1.4643	59.6	287.15	Black	Slight reduction	.035	Tallow stearine.	85.00
Means	.9095	38.1	40.6	37.4	1.4651	57.9	279.40193	85.31

NOTES ON TABLE NO. 22.

This table presents the results of an interesting study of compound lards in which the natural hog grease is reduced to a minimum.

Indeed it appears from the general results in some of the samples that they may not have any lard in them at all, but lard stearine instead. The high specific gravity, low melting point, high refractive index, great rise of temperature with sulphuric acid, and high iodine number, all point to samples containing a maximum quantity of cotton oil or cotton-oil stearine.

The chief variations from the mean of the specific gravity are shown in Nos. 5569 and 5635. In both cases the iodine numbers conform to the indications of the density.

In one case (No. 5573) the melting point is very low, while the highest melting point is (5638) a compound lard made for the Cuban trade, and having, therefore, presumably a large content of oleo-stearine, and in which we might expect the cotton oil to be present as a stearine also.

The refractive indices reveal unmistakably the presence of a body with a higher index than pure lard, and the high temperatures reached with sulphuric acid are a further evidence that this substance is cotton oil. The iodine numbers furnish the rest of the evidence, showing the high percentage of this substance present in the mixture.

The results shown in this table are much more satisfactory than those recorded in the preceding one. It is quite evident that some samples of Armour's lards might pass as pure hog grease, when the microscope fails to reveal crystals of beef fat, while none of the Fairbank samples could be thus mistaken.

Miscellaneous Lards.

- No.
- 5551. "Cream Leaf Lard," brand of Charles G. Kriel; purchased in Washington, D. C. Affidavit of Walter L. Hill.
 - 5556. "Choice Family Lard," brand of Kansas City Packing Company, Kansas City, Mo.; purchased in Mobile, Ala. Affidavit of F. H. McLarney.
 - 5560. "Choice Refined Family Lard," brand of Jacob Dold & Co., Kansas City, Mo.; purchased in Macon, Ga. Affidavit of T. Skelton Jones.
 - 5568. "Choice Family Lard," brand of Fowler Brothers, Chicago, Ill.; purchased in Saint Louis, Mo.
 - 5571. "Cream Leaf Lard," brand of Charles G. Kriel, Baltimore, Md.; purchased in Saint Louis, Mo.
 - 5575. "Choice Family Lard," brand of Anglo-American Provision Company, Omaha, Nebr.; purchased in Atlanta, Ga.
 - 5577. "Choice Family Lard," brand of George Fowler, Kansas City, Mo.; purchased in New Orleans, La. Affidavit of E. K. Converse.
 - 5578. "Choice Refined Family Lard," brand of Allcutt Packing Company, Kansas City, Mo.; purchased in New Orleans, La. Affidavit of E. K. Converse.
 - 5579. "Choice Family Lard," brand of Kansas City Packing Company, Kansas City, Mo. Affidavit of E. K. Converse.
 - 5580. "Pure Refined Family Lard," Plankinton Company brand, Milwaukee, Wis.; purchased in New Orleans, La. Affidavit of E. K. Converse.

5585. "Anchor Lard," brand of A. H. Worthman & Co., Philadelphia, Pa.; purchased in Norfolk, Va. Affidavit of C. A. Woodard and W. B. Pearman.
5587. "A No. 1 Refined Lard," brand of Swift & Co., Chicago, Ill.; purchased in Norfolk, Va. Affidavit of C. A. Woodard and W. B. Pearman.
5588. "Pure Refined Lard," brand of Chicago Packing and Provision Company; purchased in Norfolk, Va. Affidavit of C. A. Woodard and W. B. Pearman.
5589. "Kettle-Rendered Leaf Lard," brand of Rohe & Bros., New York; purchased in Norfolk, Va. Affidavit of C. A. Woodard and W. B. Pearman.
5590. "Choice Family Lard," purchased in Boston.
5594. Brand: "Pure Family Lard," Halstead & Co., New York. Affidavit of W. L. Hill.
5597. Brand: "(Marca Castellana) Mantica pura Calla Forsyth," New York. Affidavit of W. L. Hill.
5598. Brand: "Choice Refined Family Lard," Allcutt Packing Company, Kansas City, Mo.; purchased in Dallas, Tex. Affidavit of Thomas F. McEnnis.
5599. "Choice Family Lard," brand of Charles F. Tietjen, from Naphy & Co., Philadelphia.
5602. Adulterated Refined Lard, purchased in Saint Louis, Mo. Affidavit of R. A. Hamilton.
5632. Received from D. E. Fox, from Charles F. Tietjen, for Central Lard Company, New York. Affidavit of Charles F. Tietjen.
5633. "Marked B, same as A," from D. E. Fox.
5667. Kettle-rendered, backs and leaf lard, from Plumb & Winter, Bridgeport, Conn.
5668. Kettle-rendered, backs and leaf lard, from F. A. Bartran & Co., Bridgeport, Conn.
5669. Kettle-rendered, intestinal and head lard, from F. A. Bartran & Co., Bridgeport, Conn.

TABLE NO. 23.—*Lards of miscellaneous origin.*

No.	Specific gravity at 35°.	Melting point, C°.	Melting point of fat acids, C°.	Crystallizing temperature of fat acids, C°.	Color reactions with—		Refractive index at 25°.	Rise of temperature with H ₂ SO ₄ C°.	Reaction with silver nitrate.		Water.	Microscopic indications.	Iodine absorbed.
					H ₂ SO ₄ .	HNO ₃ .			Bechi.	Milliau.			
5551	.9063	37.7	38.3	1.4329	45.5	Light red	<i>Per ct.</i> .035	<i>Per ct.</i> 66.42
5556	.9057	40.0	43.2	39.9	1.4619	272.43	Marked brown	Slight reduction.	2.080	Lard stearine	62.09
5560	.9063	42.7	44.0	41.0	1.4723	50.6	Slightly brown	No reduction	.070	Tallow stearine	57.18
5568	.9072	42.7	42.2	39.6	Brown	Red-brown	1.4635	29.5	Dark red-brown	do	.621	do	66.34
5571	39.1	39.2	37.0	Very deep brown	Muddy brown	1.4615	Red-brown, doubtful.155	Lard and tallow stearine.	61.58
5575	.9051	44.5	Pink, then brown	Muddy pink	1.4608	37.7	Brown-black, light-green tint.130	Tallow stearine	61.76
5577	.9056	40.2	Faint muddy pink,	Marked pink	1.4613	44.9	Light brown, doubtful.335	Lard and tallow stearine.	76.91
5578	.9071	40.4	42.0	39.6	Pink brown	1.4665	50.1	Light brown	1.565	do	69.67
5579	.9067	36.0	40.6	37.1	Light red-brown	Light red	1.4641	47.5	Brown-black	2.110	do	62.17
5580	.9061	44.1	45.2	40.1	Red-brown	Dirty red-brown	1.4618	43.4	Brown-black, green tint.020	Tallow stearine	59.69
5585	Trace	Trace	Light red	17.385	Lard, and trace of tallow stearine.
5587	42.9	Light brown	Pink, then red-brown.	Brown-black, green tint.080	Tallow stearine	58.89
5588	45.2	Dark brown	Brown-red	1.4673	do145	do	55.88
5589	Light pink-brown.	Pink, then red-brown.	do	1.140	do	61.92
5590	.9060	40.7	44.4	39.5	Light dirty-brown.	Light red-brown	1.4612	41.8	do	Marked reduction.	do	63.61
5594	.9075	42.4	39.7	Dirty brown	Marked yellow-red.	1.4629	50.3	do	do	1.390	do	74.28

TABLE NO. 23.—*Lards of miscellaneous origin*—Continued.

No.	Specific gravity at 55° C°.	Melting point of fat acids, C°.	Crystallizing temperature of fat acids, C°.	Color reactions with—		Refractive index at 25°.	Eise of temperature with H ₂ SO ₄ C°.	Saponification equivalent.		Reaction with silver nitrate.		Water.	Microscopic indications.	Iodine absorbed.
				H ₂ SO ₄ .	HNO ₃ .					Bechi.	Milieu.			
5597....	.9072	44.9	40.7	Dirty brown....	Light red-yellow.	61.3	Brown-black, green tint.	Slight reduction.	<i>Per ct.</i> .130	Tallow stearine	<i>Per ct.</i> 69.57
5598....	.9053	42.3	40.4	Light red-brown	Dirty light brown.	1.4616	42.8	do	1.335	do	65.45
5599....	.9069	42.8	39.5	Light brown....	do	1.4638	51.1	271.82	do	do	35.661	Lard and tallow stearine.	59.62
5602....	Trace of color	Decided red	Black.	do	73.02
5632....	.9059	42.5	40.9	Marked brown.	Pink, then red	1.4614	49.4	277.14	Brown-black, green tint.	Slight reduction.	.685	Tallow, and probably lard stearine.	68.29
5633....	.9066	42.3	37.9	Marked brown	do	1.4630	49.0	276.37	Very brown-black, green tint.	do	.650	Lard and tallow stearine.	68.35
5667....	.9045	42.3	40.8	Trace.....	Trace.....	1.4610	41.7	278.86	Light red	Trace of reduction.	.065	Lard stearine...	67.27
5668....	.9046	38.9	39.5	do	do	1.4618	42.3	do	do	.110	Lard and trace of tallow stearine.	52.79
5669....	.9049	41.4	40.4	do	do	1.4628	33.6	277.95	do	do	.130	Lard stearine...	61.45
Means	.9067	41.7	39.6	1.4633	45.7	274.67	2.870	64.34

NOTES TO TABLES Nos. 23, 24, AND 25.

In Table 23 are included all the lards of a miscellaneous origin which have been examined by us, and which could not be properly classified under any of the preceding heads. Of these quite a number deport themselves with re-agents as pure hog grease, while others are without doubt adulterated. Of those which appear pure I will mention Nos. 5551, 5667, and 5669.

The microscope revealed the presence of beef fat in most of the other samples, while other tests confirmed the presence of adulterants.

In Table 24 are collected the data obtained by the analyses of crude cotton oils and foots or tank settlings.

Nos. 5603 and 5604 were furnished as pure cotton oils, but the analyses showed that they were heavily adulterated. These are the only instances in which crude or refined cotton oils were found to be adulterated. The nature of the adulterant was not determined.

Table No. 25 contains the analyses of miscellaneous oils, and especially of dead-hog grease.

The low specific gravity of the lard oil (No. 5621) appears anomalous, since it should be higher than pure lard. Further investigations will determine the normal density of lard oil at any given temperature. Pea-nut oil (No. 5622) has practically the same specific gravity as cotton oil; while olive and rape seed oils have densities slightly above pure lard. Dead-hog lard differs from the pure variety chiefly in its refractive index and the quantity of free acid it contains.

Crude cotton oils and foots.

No.

5570. Cottonseed foots, obtained from Henry Sayers & Co., Saint Louis, Mo.

5582. Crude cotton oil, purchased in New Orleans, La.

5583. Cotton-oil foots, purchased in New Orleans, La.

5603. Summer white cotton oil, from Francis Whittaker & Sons, Saint Louis, Mo.

Affidavit of R. A. Hamilton.

5604. Yellow cotton oil; source and affidavit as above.

5605. Crude cotton oil; source and affidavit same as above.

5614. Crude cotton oil; received from D. E. Fox.

5682. Crude cotton oil; from Southern Cotton Oil Trust.

5687. Crude cotton oil, from Brinkley, Ark., obtained from David Wesson, Chicago, Ill.

5688. Crude cotton oil, from Jackson, Tenn.; obtained from David Wesson, Chicago, Ill.

TABLE NO. 24.—*Crude cotton oils and foots.*

No.	Specific gravity at 55°.	Melting point, C°.	Melting point of fat acids, C°.	Crystallizing temperature, acids, C°.	Color reactions with—		Refractive index at 25°.	Rise of temperature with H ₂ SO ₄ , C°.	Saponification equivalent.	Reaction with silver nitrate.		Water.	Iodine absorbed.
					IL ₂ SO ₄ .	HNO ₃ .				Beeli.	Milliau.		
5570	43.6	43.6	Dirty yellow-brown.	Dirty yellow	1.4613	Very black	Per cent. 33.115	Percent. 110.69
5582	.9137	43.2	35.6	Deep red-brown...	Red brown	1.4674	77.2	Very red, n black.120
5583	.9154	39.9	do	Marked red.	1.4679	77.7	Deep red	2.850	108.33
5603	43.6	Deep black	Very brown-black.	Marked red.	No reduction	.795	73.92
5604	38.4	34.1	Dark brown	Dark brown	Dark brown-black	Trace of reduction.	89.81
5605	Deep brown-red.	Deep brown red.	1.4618	Deep red, but not black.100	109.96
5614	.9167	38.4	35.1	Deep black-brown	Marked red-yellow.	1.4637	73.8	Black-brown, green tint.135	104.25
5682	.9164	40.2	34.9	1.4670	79.0	281.81	Very black	109.45
5687	.9151	Deep red brown	107.76
5688	.9144	43.2	33.6	1.4665	Dark red-brown.	Slight reduction.	109.67
Means	.9156	43.6	41.3	34.7	1.4655	76.9	281.81	6.186	102.65

Miscellaneous oils.

No.

5617. Marked "Olive Oil Sublime," from Z. D. Gilman, Washington, D. C.
5620. Rape-seed oil, from Z. D. Gilman.
5621. Lard oil, from Z. D. Gilman.
5622. Pea-nut oil, from Z. D. Gilman.
5623. Labeled "Hughes's Extra Superfine Italian Lucca Olive Oil; purchased from Alfred E. Hughes, Boston, Mass. Affidavit of Walter L. Hill.
5624. Olive oil, purchased from Wm. Underwood & Co., Boston, Mass. Affidavit of Walter L. Hill.
5627. Olive oil from Alden Speare's Sons & Co., Boston, Mass. Affidavit of Walter L. Hill.
5651. "Dead-hog grease," from John P. Squire & Co., Boston, Mass.
5659. "Dead-hog grease," from East Saint Louis Rendering Company, East Saint Louis, Ill. Affidavit of R. A. Hamilton.
5660. Dead-hog grease, with 10 per cent. of oleo-stearine added, from East Saint Louis Rendering Company, East Saint Louis, Ill. Affidavit of R. A. Hamilton.
5670. Dead-hog grease from East Saint Louis.
5671. Dead-animal grease from East Saint Louis.
5689. Prime lard oil from David Wesson, Fairbank & Co., Chicago, Ill.

TABLE NO. 25.—*Miscellaneous oils.*

No.	Specific gravity at 33°.	Melting of fat acids, C°.	Crystallizing temperature, acids C°.	Color reactions with—		Refractive index at 25°.	Rise of temperature with H ₂ S O ₂ , C°.	Reaction with silver nitrate.		Water.	Iodine absorbed.
				H ₂ SO ₄ .	HNO ₃ .			Bechi.	Millian.		
5617....	.9076	26.6	19.55	Brown-black.....	Yellow-green.....	1.4642	50.9	Dirty red-yellow....	No reduction.....	Per cent.	Per ct.
5620....	.9059	21.6	do.....	Light red.....	1.4672	68.0	Light dirty brown....	do.....	.035	98.72
5621....	.8949	42.6	Deep brown.....	Deep dirty-red....	1.4632	54.2	Light brown.....	do.....	1.365	66.36
5622....	.9124	32.6	30.87	1.4638	69.1	do.....	.125	96.61
5623....	.9076	Green, changing to brown.	Light green.....	1.4643	Light green-brown..	83.38
5624....	.9080	24.2	17.27	do.....	do.....	1.4638	49.0	Light green-yellow..	No reduction.....	81.90
5627....	.9039	27.0	Deep black-brown..	Light yellow.....	1.4639	53.0	Light red-brown....	Mere trace.....	.100	82.76
5651....	.9081	41.4	38.90	Black-brown.....	Dirty brown.....	1.4615	44.0	Light red.....235	61.70
5659....	.9086	42.0	37.10	Light pink-brown..	Decided pink.....	1.4618	42.4	No color.....	Jelly.....	.155	60.52
5660....	39.60	do.....	Marked pink.....	do.....045	56.13
5670....	.9058	40.4	38.50	1.4664	43.7	do.....	Mere trace.....	.245	65.49
5671....	.9037	42.6	40.45	1.4585	37.5	Faint yellow.....	No reduction.....	3.780	53.42
5689....	.9151	1.4650	No color.....	85.63
Means	1.4638	51.2676	75.23

ABSTRACTS OF METHODS USED IN LARD ANALYSIS, WITH RESULTS THEREOF, RECENTLY USED BY ANALYSTS.

Dr. Shippen Wallace* has made a study of the adulteration of lard. His conclusions are as follows:

In all samples of suspected lard, if one will follow the method here given, he can not fail to meet with correct and proper results.

(1) Hübl's method, which will indicate either adulteration with tallow alone or cotton-seed oil alone, or indicate pure lard.

(2) Use Bechi's test, as described, which will prove the presence or absence of cottonseed oil.

(3) Use the sulphuric-acid test as a further confirmation.

By these last two, if Hübl's method should yield a figure which would classify the suspected lard as pure, one can readily confirm or disprove it, while if Hübl's should indicate cottonseed oil, they would make the proof complete. Lard stearine yields figures, by Hübl's method, within the range of pure lard, and while some manufacturers make use of this article in the manufacture of summer lard, yet it is not an adulteration in the same sense that cottonseed oil and tallow are. I have not mentioned other claimed adulterants of lard, as they are easy of detection; whatever we sometimes find, one sample I examined containing 11.80 per cent. When this is found it is either caused by carelessness in the manufacture, or is intentional, as it can readily be guarded against.

The percentages of iodine absorbed by sixteen samples of pure and adulterated lard as found by Dr. Wallace are given in the following table:

No.	Pure.	Adulterated.	No.	Pure.	Adulterated.
	<i>Per cent.</i>	<i>Per cent.</i>		<i>Per cent.</i>	<i>Per cent.</i>
1	57.9	58.0	9	61.0	70.2
2	59.6	53.4	10	59.4	67.0
3	59.2	58.1	11	59.5	69.4
4	60.3	60.4	12	59.0	65.1
5	57.7	59.5	13	58.9	63.8
6	61.7	61.8	14	59.1	70.7
7	62.2	60.0	15	60.0	72.0
8	57.4	68.6	16	60.4	66.8

NITRATE OF SILVER REACTION WITH COTTON OIL.

Bizio† criticises the report of the Italian commission which recommended Bechi's test for detecting cotton oil.

According to Bizio pure olive oil sometimes produces reduction of silver even when the re-agent is slightly acidified with nitric acid. On the other hand, some samples of cotton oil fail to produce the reduction. Bizio did not take the same care to identify his samples that was used by the commission, and his criticism will not impair the value of the large experience which has shown the practical reliability of the silver test in the detection of cotton oil.

* Report of the Dairy Commissioner of the State of New Jersey for 1887, p. 16 *et seq.*

† Chem. Central-Blatt, June 23, 1888, p. 873.

CHLORIDE OF GOLD AS A TEST FOR COTTON OIL.

Hirschsohn* has recommended the use of aurous chloride for the detection of cotton oil. The reagent is used as follows:

Dissolve one gram of gold chloride in 200cc of chloroform. To 3 to 5cc of the oil add 6 to 10 drops of the re-agent and heat for twenty minutes. Cotton oil will give a beautiful red color.

David Wesson† finds that free fatty acids interfere with the delicacy of the reaction, and also rancid lard.

Free acids and rancid lard, on the other hand, do not affect the process of Brullé.

Moerk‡ has also reported results of this test.

I have tried the reaction of Hirschsohn, and found the purple color produced quite characteristic; but even pure lards give a trace of color, which must not be confounded with the deep coloration produced by cotton oil.

BRULLÉ'S METHOD OF DETECTING ADULTERATIONS IN OLIVE OIL. §

The United States consul (Mr. Mason) at Marseilles writes as follows:

Southern France has of late years suffered seriously from the adulteration, or rather the artificial fabrication, of her two principal agricultural products, wine and olive oil. During the recent season of scanty vintages there has grown up in this district an immense manufacture of "piquettes" or raisin wines, which are made by soaking in water, until fermentation takes place, the cheap dried grapes which are imported in such quantities from the Grecian Archipelago and Turkey. These substitutes have so far replaced the real but more costly French wines that now—since the replanted vineyards begin to yield more abundantly—the genuine ordinary wines command only prices which hardly repay the cost of culture. The consumption of vinous beverages among the laboring classes has not diminished, but the cheaper substitute has crowded out the real article, and in behalf of the agricultural class it is proposed to remedy this unnatural difficulty by putting a heavy import duty upon dried grapes from the Levant.

With olive oil the case is similar, but even worse. Only a small portion of France is adapted to olive culture, the entire available district being a strip of dry country less than 20 miles wide along the Mediterranean coast. The tree is of slow growth, and is, moreover, beset by numerous insects and diseases, which, in addition to unfavorable phases of weather, render the yearly olive crop more or less uncertain. Any serious reduction in the annual consumption of olive oil is sufficient to

* Pharmaceutische Zeitschrift für Russland, 1888, p. 721, and American Journal of Pharmacy, January, 1889, p. 23.

† Letter February 16, 1889.

‡ American Journal of Pharmacy, February, 1889, p. 65.

§ The Grocer and Oil Trade Review, February 2, 1889.

reduce its market value below the point of profitable culture. This has been done by the now nearly universal practice of adulterating or diluting the olive oils of Nice and Provence with various seed oils, viz, sesame, peanut, poppy-seed, camomile, and especially cottonseed, which last, by reason of its cheapness, palatable flavor, and difficulty of detection, has of recent years nearly supplanted all the others as an adulterating material. The rank, low-priced olive oils from southern Italy (Bari), Algeria, and Tunis have been brought here in vast quantities, diluted with cotton or sesame, and been consumed and exported wholesale in place of the fine, delicate, high-grade oils of the Var and Bouches-du-Rhône, which have thus been nearly elbowed out of the market. This has so reduced the value of olive oil in southern France that the Government has set itself seriously to the task of providing a remedy. The first step was to discover some method of detecting such adulterations which should be not only exact in its results but sufficiently simple to be practicable for farmers, dealers, and ordinary consumers. It was stated in a report which was made from this consulate in February, 1888, that no such process was then known. As late as May 17 last a meeting of the Scientific and Industrial Society of Marseilles was addressed by Mr. Ernest Millian, an accomplished analytical chemist, who reviewed elaborately all of the known processes and admitted that none of them were sufficiently delicate and exact to detect an adulteration of less than 10 per cent. The "Cailletet" process, which consists in treating the oil with a mixture of sulphuric and nitric acids, had been hitherto generally employed, but this was declared by Mr. Millian untrustworthy unless the degree of adulteration exceeded 20 per cent.

The "Bechi" process, now used by the Italian Government, will detect an admixture of 15 per cent. of cottonseed oil, provided the sample analyzed contains no glycerine, formic acid, or free fatty acids, any one of which, even in minute quantity, is sufficient to mask the chemical reaction upon which the process of Signor Bechi depends. Mr. Millian then described a new method, invented by himself, which consists in treating with heat the saponified products of the oil in alcoholic solution with nitrate of silver. This, however, is a process for the laboratory of the accomplished chemist, and is not adapted to general use. The same is true of the "Levallois" process, which has been used by experts in cases of real importance with more or less questionable results, the analysis in one notable instance having given the same result from a sample of pure olive oil, and another which was known to contain 20 per cent. of cotton-seed.

Finally, as it would seem, the long sought for process has been discovered by Mr. Brullé, chemist of the Agronomic Station at Nice. His discovery was announced to the Academy of Sciences in April last, and has been since subjected to an elaborate series of tests and experiments by a commission specially appointed for the purpose by the Ag-

ricultural Society of the Alpes Maritimes. Mr. Brullé began upon the known principle that vegetable oils, when oxidized by the application of certain acids, assume different shades of color. He then hit upon the use of albumen to fix and accentuate these delicate gradations of tint. The report of the commission has recently been published, and gives the process of Mr. Brullé such complete and unqualified indorsement, both for its simplicity and the exactness of its results, that the subject assumes a practical importance not only to the countries which produce olive oils, but to those which, like our own, import them as costly luxuries for general consumption. In its series of experiments at Nice the commission first applied the process of Mr. Brullé to six classes of samples, viz, first, to pure olive oil, then to the same oil with an added admixture of 5, 10, 20, and 50 per cent., respectively, of cottonseed oil, and finally to the pure cottonseed oil itself. When the result had been established by repeated experiments with each grade of samples a fac-simile of the tint produced by each successive degree of adulteration was prepared by dissolving certain pigments in stated quantities of water. Thus the process and a standard system of proofs were put within reach of any person having a good eye for color and a slight familiarity with chemical manipulations.

THE NEW PROCESS.

The process of Mr. Brullé is as follows: Put into a test-tube $1\frac{1}{2}$ grains* of pure albumen (this should be gently heated in the flame of an alcohol lamp to expel any remaining moisture in the albumen which might otherwise modify the exactness of the result), then add 3 cubic centimeters of nitric acid and 10 cubic centimeters of the oil to be tested (the quantity of each ingredient used is, of course, immaterial, provided the above relative proportions are maintained; a test-tube graduated metrically is the most convenient for the purpose); the mouth of the tube is then closed with a cork to prevent the boiling over of the liquid during ebullition, but pierced with a small orifice to permit the escape of vapor, which would otherwise explode the tube. The materials are mixed by shaking, but the nitric acid quickly settles to the bottom. Now warm gently in the lamp the part of the tube containing the oil, then apply the flame to the underlying stratum of acid. A fierce ebullition soon ensues, and when this is at its height plunge the tube into ice water sufficiently cold to chill the contents to 4° C, or its equivalent 40° F. During the cooling process there is developed an oleaginous precipitate, ranging in color from pale yellow to reddish brown, according to the proportion of cotton oil contained in the tested sample. The experiment requires only the simple apparatus above mentioned, and occupies only four or five minutes.

The findings of the commission at Nice are tabulated in its official

* 50 mg.

report as follows, the standard tint in each grade being produced by dissolving the stated number of units of each pigment named in 100 units of water. For this purpose ordinary dry-cake water colors are most convenient:

(1) Pure olive oil yields a precipitate tinged like 5 units of Naples yellow dissolved in 100 units of water.

(2) Olive oil containing 5 per cent. of cotton oil yields the tint of 5 units Naples yellow and 5 units of dark chrome yellow in 100 units of water.

(3) Olive oil containing 10 per cent. cotton seed yields a tint equal to 20 units Naples yellow, $6\frac{1}{2}$ units chrome yellow, and 1 unit Chinese vermilion in 100 units of water.

(4) Olive oil containing 20 per cent. cotton seed yields a tint equal to $6\frac{1}{2}$ units Naples yellow, 6 units chrome yellow, and $1\frac{1}{2}$ units Chinese vermilion similarly dissolved.

(5) Olive oil with 50 per cent. cotton oil yields a tint equal to 5 units Naples yellow, 5 units chrome yellow, and 5 units of vermilion.

(6) Pure cotton oil yields a precipitate having the color of $3\frac{1}{2}$ units chrome yellow, 10 units of vermilion, 1 unit of burnt sienna, and 1 of natural sepia in 100 units of water.

Other seed oils, including sesame, camomile, peanut, and poppy seed, give a precisely similar series of tints in proportion to the degree of their admixture with olive oil, except that the colors are more inclined to the reddish shade which would be produced by covering the corresponding cotton-seed tint with a thin wash of carmine. These gradations of color are most marked when the liquid in the tube is at about the stated temperature, 40° F. As the precipitate is further chilled to the freezing point the colors fade and lose their individuality. Such is the system which is now expected will enable purchasers and consumers of olive oil in this country to detect the adulterations, which have become so general that very few brands or firm names are any longer a guaranty of purity. When it is remembered that more than 2,000,000 gallons of cotton-seed oil are exported from the United States to Marseilles in a single year, and that more than half of this vast quantity is used for adulterating olive oils, a large part of which are re-imported to the United States through a 30 per cent. duty, the importance of some new and better means of controlling the integrity of this trade will be apparent. Some time ago 1,000 tierces of American lard were stopped at the wharf in Marseilles, and the consignees subjected to a costly process, which is not yet terminated, because the lard was found upon analysis by the customs officers to contain 10 per cent. of cottonseed oil. This seizure was based upon the fact that, while lard is entitled to entry duty free, cottonseed oil bears a duty of 6 francs per 100 kilograms, and this adulteration of a free article with a dutiable one is held to be fraudulent. The least that can happen to the shippers in this case will be that they must pay the duty on 100 tierces of cottonseed oil and

the expenses of the process, besides the loss which the consignee suffers from the delay. Might not this rigid scrutiny be equally well applied to some of the adulterated and falsified foreign products which are landed at American ports?

It is not within the scope of this report to consider whether either lard or olive oil, when adulterated with cottonseed, is necessarily unwholesome. The vital fact is that in paying from 40 to 50 cents per kilogram and 30 per cent. duty on American cottonseed as olive oil, the people of the United States are submitting to a wholesale fraud, the proportions of which are increasing year by year.

The interest of both the United States and France will be subserved when the reckless tampering with the integrity of commerce is systematically suppressed. As long as our people will accept and pay for adulterated oils they will continue to flood and dominate the market. The remedy must be applied at our ports of entry.

Mr. David Wesson* makes the following comments in regard to Brullé's and other methods of testing for cotton seed oil:

"We have worked some with the chloride of gold test and find it will give a reduction with cottonseed oil, free fatty acids, and old rancid lard. It gives no reduction with pure fresh lard containing less than 1 per cent. of free acid.

"We find the Brullé test is unaffected by free acid or rancidity. We have tried the Bechi test on some highly oxydized cotton oil and find it gives no reduction whatever; while with lard oil made from old lard considerable reduction can be obtained."

COCOA-NUT OIL AS AN ADULTERANT OF LARD.

It is probable that in this country lard is never adulterated with cocoa-nut oil for commercial purposes. Allen† speaks of the use of cocoa-nut oil as an adulterant of lard. In "The Analyst," October, 1888, page 89, he says he is unable to trace the authority on which the statement was made. He has, however, in his own experience found one sample of lard which was adulterated with cocoa-nut oil. This lard gave the following numbers on analysis:

Specific gravity at 99°8666
Iodine absorption, per cent.	37.4
Saponification equivalent	265.2
N	
$\frac{10}{10}$ alkali for the distillate from 2½ grams ..	3.3cc.

The volatile acids contained a notable proportion of soluble acids of sparing solubility in water, and had the characteristic odor of the distillate from cocoa-nut oil. The sample was certified to contain 33 per cent. of the adulterant. The most accurate determination of the cocoa-nut oil is obtained from the saponification equivalent. Mr. Allen gives

* Letter of March 4, 1889.

† Commercial Organic Analysis, Vol. 2, p. 142.

the saponification equivalent of lard at 289 and cocoa-nut oil at 219; hence every .7 fall in the equivalent below 289 indicates the probable presence of 1 per cent. of the adulterant. Pressing inquiries have been sent to Mr. Allen from America as to where cocoa-nut stearine could be obtained, but none was found to be on the market. The comparison of the analyses of pure lard and cocoa-nut oil is given in the following table:

	Lard.	Cocoanut oil.
Original fat:		
Plummet gravity at 99° C.....	.860 to .861	.868 to .874
Iodine absorption.....	55 to 61	9
Saponification equivalent.....	286 to 292	209 to 228
Volume of $\frac{N}{10}$ alkali required by distillate from 5 grams.....	0.5	7.0
Separated fatty acids:		
Plummet gravity at 99° C.....	.838 to .840	.844
Iodine absorption.....	61 to 64	15.61
Mean combining weight.....	278	200

DETECTION OF COTTONSEED OIL IN LARD.

Mr. A. H. Allen* has made a further study of the detection of cotton-seed oil in lard. As a result of his analyses he gives the following figures:

	Omentum lard.	Leaf lard.	English lard.	American lard containing cottonseed oil.	Mixture of unknown nature.	Suspected sample.
Original fat:						
Melting point, C°.....	39.0	40.0	39.0	37.5	40.0
Solidifying point, C°.....	32.5	32.0	27.0	27.5	30.5
Plummet gravity at 99 C°.....	.8602	.8620	.8608	.8648	.8637	.8637
Iodine absorption, per cent.....	55.4	60.5	62.0	82.5	68.8	62.8
Fatty acids:						
Melting point, C°.....	39.0	39.5	39.5
Solidifying point, C°.....	38.7	38.5	37.5
Plummet gravity at 99 C°.....	.8372	.83858450	.8385
Mean combining weight.....	274.5	276.8
Iodine absorption, per cent.....	58.3	65.3	70.4	64.8
Oleic acid, etc., per cent.....	58.4	57.8
Oleic acid, iodine absorption.....	87.4	(91.6)
Milliau's nitrate of silver test.....	White.	White.	Gray.	Marked blackening.	Marked blackening.	Sensible darkening.

¹ Rising to 27.5² Rising to 39.0.³ Rising to 38.8.⁴ Rising to 38.5.

*The Analyst, September, 1888.

He also gives the result of a comparison of tallow, lard, and cotton oil :

	Tallow.	Lard.	Cotton oil.
Original fat:			
Melting point, C°.....		28 to 45	Fluid.
Solidifying point, C°.....	33 to 48		
Specific gravity at 99 C°.....	.862	.860 to .861	.872
Iodine absorption, per cent.....	40	59 to 62	165 to 110
Fatty acids:			
Melting point, C°.....	45	38	25 to 36
Solidifying point, C°.....	43	38	30
Specific gravity at 99 C°.....			.8467
Iodine absorption, per cent.....	41.3	64.2	115.7

Analyses were also made of cotton oil and cotton-oil acids, as indicated in the following table :

	A.—Cotton oil stearine	B.—Cotton oil.	C.—Cotton- oil acids from B.
Plummet gravity at 99 C°.....	.8684	.8725	.8476
Melting point C°.....	40		35
Solidifying point C°.....	* 31		32
Iodine absorption.....	89.8	108 to 110	115.8
Saponification equivalent.....		285 to 294	289
Acidity (=oleic acid).....	.34	Trace.	97.6

* Rising to 32.5°.

It is found that there is a marked difference in the specific gravity of lard and cotton oil, and also in the iodine absorption of the two. Lard and beef fat have substantially the same specific gravity. The difference is important, since it would enable one to distinguish a mixture of beef stearine and cotton oil, having an iodine absorption of about 60 from genuine lard. Thus, with a proportion of the adulterant in a mixed composition of lard, the cotton oil only can be ascertained with considerable accuracy by determining the iodine absorption; the estimation will be below the truth if beef stearine be present. On the other hand, the presence of beef stearine does not interfere with the deduction to be drawn from the increased specific gravity of the melted sample.

Mr. Allen finds Milliau's nitrate of silver test to be valuable, and prefers it to the original one proposed by Bechi. In his opinion the indications obtained from the melting-point or solidifying-point of the glycerides of the fatty acids are of no value. Samples of lard oil were found to have an iodine absorption of 73 and 74, while one several years old gave only 41. It is recommended that the iodine absorption be determined on the fatty acids instead of the original glycerides, thus avoiding the use of chloroform, which has a marked disturbing influence on the strength of the iodine solution employed.

Hehner* states that in Bechi's test, without impairment of the delicacy, the re-agent may be made up without the amyl alcohol or rape-seed oil. He makes the solution of nitrate of silver in alcohol and ether very slightly acidified, and adds to the oil to be examined about one-half of the bulk of the silver solution, and then heats on the water-bath for one-quarter of an hour longer. Pure lard always remains perfectly unchanged by this treatment, while cotton-oil mixtures blacken more or less quickly. It is quite possible to arrive at approximate quantitative results by comparing the oil mixtures of known composition. Mr. Hehner does not see any advantage in Milliau's modification. The rise of temperature when mixed with sulphuric acid is to be preferred as a method of estimating the quantity of cotton oil in lard. The sample, of course, must be free from water. When 50 grams of pure lard, according to Hehner, are mixed with 10cc of strong sulphuric acid the rise of temperature varies from 24° to 27.5° , while cotton oil in the same conditions shows an increase of 70° . In every case lard which reduces silver shows an increase of temperature of more than 27.5° .

[NOTE.—Compare these temperatures with those obtained in our experiments. The mean rise of temperature for pure lard was 41.5° , and the mean increase for cotton oil 85.4° .]

Roland Williams† has also contributed a study to the adulteration of lard with cotton oil. He regards the saponification equivalent as quite useless as far as the detection of cotton oil in lard is concerned, as both the lard and cotton oil require practically the same amount of alkali for saponification. In case of the use of cocoa-nut oil, however, the determination of the saponification equivalent is of the highest importance.

The melting-point also is regarded as of no value in respect of the detection of adulteration, since it depends largely on the parts of the animal from which the fat has been obtained. The specific gravity of pure lard at the boiling-point of water is about .861, and of cotton-oil at the same temperature .872. It may be possible, therefore, to derive some valuable information in regard to the constitution of lard or mixed lards from a careful determination of the specific gravity. Mr. Williams failed to obtain valuable results with Maumené's test. This failure was doubtless due to some imperfection in the method of manipulation.

In the absence of interfering bodies Mr. Williams relies chiefly upon the percentage of iodine absorbed in estimating approximately the amount of cotton oil present as an adulterant. The addition of stearine to lard interferes seriously with the determination of the percentage of added cotton oil by the iodine method. He has found pure lards to absorb from 60 to 62 per cent. of iodine. One sample of lard, said to be leaf lard, absorbed only 51 per cent. Some leaf lard rendered by Mr. Allen himself absorbed 51.8 per cent.

* The Analyst, September 1888, p. 165, *et seq.*

† The Analyst, September, 1888, pp. 168, 169.

Milliau's modification is recommended, but it is advised that a blank experiment be made with the re-agent, since sometimes alcohol contains impurities which reduce silver nitrate. Experiments in the use of the silver nitrate test for quantitative purposes did not give satisfactory results.

Jones* says that he was the first public analyst of England to certify a case of lard adulterated with cotton oil under the sale of food and drugs act. He first applied a qualitative test with chloride of sulphur essentially the process described by Warren. He used 5 grams of the fluid lard in a porcelain dish, to which he adds 2cc of equal volumes of chloride of sulphur and bisulphide of carbon. The mixture is well stirred at first and occasionally for fifteen or twenty minutes. No heat is applied. By this treatment genuine lard only thickens or becomes rather stiff in three hours. If it contain cotton oil it becomes quite hard and solid in one-half hour. This test is very simple, but with practice one can with certainty pick out all lards containing cotton oil. He estimates the extent of the adulteration by the percentage of iodine absorbed. He finds that pure lard never takes sensibly more than 60 per cent. of iodine, while cotton oil takes 105 to 110 per cent. He adopts the formula—

$$100 \left(\frac{\text{I. absorbed} - 60}{45} \right) = \text{per cent. cotton oil.}$$

The accuracy of the work is checked by the specific gravity taken at 100° F. At this temperature the specific gravity of pure lard is taken at .9060, and of cotton oil at .9135.

The formula for calculating the percentage of adulteration by the specific gravity is as follows:

$$100 \left(\frac{\text{Sp. gr. found} - 906}{7.5} \right) = \text{per cent. cotton oil.}$$

The radical error in the method of Mr. Jones is, that he takes no account whatever of the admixture of stearine with adulterated lard, which may be done so skillfully as to wholly vitiate the method employed for determining the amount of adulteration.

Stock† describes a modification of Milliau's method for the detection of cotton oil. His method is as follows:

Fifteen grams of the sample are saponified in a 7 inch porcelain basin with a mixture of 15cc of 30 per cent. NaHO and 15cc of 92 per cent. alcohol. To commence, the fat is heated to 110° C. The alkaline alcohol must be added in quantities not exceeding 1cc at a time, the

* The Analyst, September 1888, p. 170.

† The Analyst, September, 1888, p. 172.

temperature not being allowed to fall below 95° C. to 100° C., constant stirring at this part of the operation being most important. If the saponification has been successful, the resultant soap is a smooth, thick paste. Boiling distilled water is now added drop by drop, a thin, flexible spatula being used to break down the paste. When this has the appearance of smooth starch, water may be run in till a volume of 500cc is reached. Complete solution should follow. Forty cubic centimeters of diluted sulphuric acid (1—10) are now added to the contents of the basin, the liquid is stirred gently and brought to boil for seven to twelve minutes, then kept just below boiling, until the separated fatty acids fuse to a clear oily layer. The greater bulk of the acid watery liquid is siphoned off, the remainder with the fatty acids being poured into a clean, warm flask with a somewhat long and narrow neck. The fatty acids are freed as nearly as possible by siphonage from the watery under layer, and the flask is filled up with boiling water so as to bring the fatty acids into the neck, by which operation a partial washing is given. Five cubic centimeters of the fused fatty acids are now transferred by means of a dry, warm, fast-running pipette, into a clean, dry, wide test tube. Twenty cubic centimeters of absolute alcohol are added, care being taken to wash the pipette by running the alcohol through it. The contents of the test tube are heated to incipient ebullition in a vessel of boiling water. Two cubic centimeters of a 30 per cent. solution of silver nitrate are now rapidly poured into the tube, when, if even 2 per cent. of cotton oil be present in the sample, the characteristic cedar-brown color is at once developed. Pure lard gives absolutely no color.

To quantify this reaction, known mixtures of pure lard and refined cotton oil are treated exactly as above, and the colors in the different tubes compared by reflected light against a white background. This must be done simultaneously, for in about seven minutes the coloring matter begins to fall out, and correct comparison is then impossible. In careful hands excellent results are obtainable.

Prof. J. Campbell Brown* calls attention to errors analysts are liable to make :

1. They are liable to underestimate the proportion of cotton oil when relying upon the iodine test alone. The reason of this is found in the admixture of stearine in adulterated lards which has a low iodine number.

2. They are liable to condemn genuine lard which is more oily than pork fat or lard rendered in England. According to Hehner American lard contains more olein than English. I do not think the assumption of Hehner a just one since the iodine number of pure lards in this country is found to be about the same as in England.

Mr. Watson Grey† gives the results of some determinations of the

* Op. cit., p. 103.

† Op. cit.

absorption of iodine by lard showing a very low absorptive power. His results are given in the following table :

Kind of lard.	Iodine absorption.
	<i>Per cent.</i>
From omentum of hog.....	49.5
Market lard (bought in Liverpool).....	49
From omentum of sow.....	55.5
From back of pig.....	65

Mr. Grey will fix the average for English lards at 57 per cent. instead of 62 as taken by Mr. Allen. Mr. Fox stated that he had recently found 50 per cent. of pea-nut oil in lard oil, determining it by the altered specific gravity and the presence of arachidic acid.

Mr. M. F. Horn* gives a method for the quantitative estimate of paraffine, cerosin and mineral oils, in fats and wax. Inasmuch as these adulterations are not likely to occur in lard I will cite only the original paper.

Roland Williams† gives a table showing the iodine numbers and melting-points of certain fatty acids. The melting-points were determined by the ordinary capillary-tube method. Following are his results :

Name of fatty acid.	Iodine absorption.	Melting-point.
	<i>Per cent.</i>	<i>°F.</i>
Tallow.....	41.3	119
Lard.....	64.2	160
Cotton oil.....	115.7	96
Olive oil.....	90.2	81
Linseed oil.....	178.5	75
Rape oil.....	105.6	71
Castor oil.....	93.9
Cocoa-nut oil.....	9.2	75
Palm oil.....	53.4	114
Sperm oil.....	88.1	56

The low melting-point in the case of lards is explained by Mr. Williams on account of the fat having been taken from the entire animal. As might be expected the fatty acids absorb a slightly greater percentage of iodine than the glycerides from which they were made.

Prof. Stephen P. Sharpless‡ relies upon the usual tests for the detection of the adulteration of lard with cotton oil. Bechi's test, he says, gives good results. Nitric acid of 1.35 specific gravity gives only a faint color with pure lard, while with lard adulterated with cotton oil it gives a color more or less intense. For the detection of added stearine made from tallow Dr. Belfield's microscopic test is employed. The suspected

* The Analyst, Oct. 1888, p. 184. Zeitschr. f. Angew. Chemie, No. 16, 1888, p. 459.

† The Analyst, May, 1888, p. 88.

‡ The Analyst, April, 1888, p. 69.

lard is dissolved in ether in a test tube, which should be about two-thirds filled. The solution should be nearly saturated. The tube is loosely stopped with cotton wool, and placed in a quiet room, at a temperature of about 60° F. When the first crystals are formed they are removed by means of a pipette, placed on the slide of the microscope, and examined in the usual way. The forms of the crystals produced have already been described.

David Wesson* says of Belfield's microscopic test, that while at times it gives very characteristic crystals, at other times their forms are not sufficiently definite to be relied upon. The nitric and sulphuric acid tests are sometimes unreliable, especially with old samples. Bechi's test is also sometimes uncertain. On old samples of cotton oil it sometimes gives negative results, while with old samples of lard oil it will give a slight reduction.

Michael Conroy† uses the following tests for the determination of the purity of a sample of lard.

(1) Heat and stir about one-half ounce of lard with one-tenth its weight of strong nitric acid, specific gravity 1.42, in a porcelain dish of about 8 ounces capacity, until a brisk action commences, when the source of heat should be removed. Pure lard sets in about one hour to a pale orange-colored solid, but if it contain cotton oil it takes a more or less deep orange-brown tint.

(2) The test of Labiche was also tried, as follows: Equal parts of the fat and neutral acetate of lead and ammonia added, stirring briskly. The acetate of lead decomposes and the nascent oxide reacts upon the oil, causing it to turn red. This reaction proved a failure.

(3) The proceeding of Ernest Milliau By this test it is claimed 1 per cent. of cotton oil can be detected.

(4) Bechi's test: When sodium carbonate has been used to correct the acidity of lard this test is not applicable, unless the reagent be acidified with nitric acid. The following modification of Bechi's test was employed: A solution of five parts of silver nitrate and one part of nitric acid, specific gravity 1.42, in one hundred parts of alcohol. Put 6 grams of lard in a dry test tube and add one-fourth gram of the solution above described, and hold the tube in boiling water for five minutes. Pure lard remains perfectly white, but if adulterated with cotton oil it assumes a more or less olive-brown color. This color is best observed when the lard sets. One per cent. of cotton oil in a lard gave a color quite distinct from the genuine article.

Cotton oil has also been used for the adulteration of tallow.‡

The melting-point of the genuine tallow, according to Williams, varies considerably in different samples, ranging from 100° to 120° F.

* The Analyst, July, 1888, p. 140.

† The Analyst, Vol. 13, No. 151, p. 203. The Pharmaceutical Journal and Transactions, September 22, 1888, p. 237.

‡ Roland Williams, Journal of Society of Chemical Industry, March, 1888, p. 186.

The best class of tallow has a melting-point of about 110° F. Pure tallow requires from 19.3 per cent. to 19.8 per cent. of caustic potash for saponification, and cotton oil 19.1 to 19.6. A series of mixtures of tallow and cotton oil was prepared containing 5, 10, 15, 20, 25, 30, and 40 per cent. of the oil. The addition of the cotton oil did not have the effect upon the melting-point which might be expected. The pure samples melted at 110° F. and the one with 40 per cent. oil at 102° F. The quantity of iodine absorbed was by the pure tallow 40.8 per cent., and by the mixture containing 40 per cent. oil 66.2 per cent. The percentages for the several samples were as follows: 44, 47.1, 49.7, 52.9, 56.1, 59.2, 66.2. The percentage of iodine absorbed by the original cotton oil was 109.1 per cent. The percentages of iodine absorption have a remarkably close connection with the percentage of cotton oil present in the various mixtures.

REACTION OF ANIMAL AND VEGETABLE GLYCERIDES FOR CHOLESTERIN AND PHYTOSTERIN.

The presence of cholesterin in animal glycerides, especially liver fat, has long been known.

A substance homologous with cholesterin was detected in the oil of Calabar beans in 1878 by Hesse, to which he gave the name of phytosterin.*

Salkowski proposes to distinguish animal and vegetable fats from each other by testing them for cholesterin and phytosterin respectively†. To obtain the cholesterin (phytosterin) 50 grams of the glycerides, animal or vegetable are saponified with alcoholic potash. The alcohol is evaporated, and the soap diluted with water to about 2 liters. This is shaken in a separating globe with ether, and the ether solution drawn off and evaporated to small bulk. The residue, which may contain a small quantity of unsaponified fat, is again treated with potash, and the separation effected by ether, as above, only a little water being added. If the ether solutions separate slowly, a few drops of alcohol may be added.

The ethereal extract is evaporated and the cholesterin separated in crystals. Animal cholesterin has a melting-point of 146° ; vegetable (phytosterin) 132° . The two also show distinctly crystalline forms which are easily distinguished under the microscope. Vegetable cholesterin shows star-shaped crystals or bundles of long, quite solid, needles, while the animal product gives thin rhombic tables.

Dissolved in chloroform, the two products show different color reactions with strong sulphuric acid. Cholesterin shows a cherry-red and phytosterin a blue-red color. In mixtures of animal and vegetable glycerides the melting-point of the cholesterin obtained may become a fair index of the proportion of the two present. Thus, a melting-point of 139°

* An. d. Chem. u. Pharm., Vol. 192, p. 178.

† Zeitschrift für Analytische Chemie, Vol. 26, p. 572.

would indicate that the fat from which the cholesterin was obtained was made up of equal proportions of animal and vegetable glycerides.

SEPARATION OF STEARINE AND PALMITINE IN LARD.

Isbert and Venator* have separated stearine and palmitine from lard in the following manner :

The sample is dissolved in cold ether in a test tube, and the closed tube allowed to stand for some time. At the end of about two hours the stearine begins to separate and is collected at the bottom of the tube. The identity of the stearine was shown by its melting-point, viz, 60° . The palmitin separates later.

The separation can also be effected by solution in boiling alcohol. The separated glycerides are separated from olein by pressing between blotting paper.

ABSORPTION OF OXYGEN.

Cotton oil absorbs a notable proportion of oxygen when subjected to the Livache process.†

Finely-divided lead is obtained by precipitating with zinc. About 1 gram of the lead powder is placed on a watch glass and mixed with nearly 5 grams of oil. The disk is placed in a well-lighted room of medium temperature.

Cotton oil gains about 6 per cent. in weight in forty-eight hours. The equivalent of oxygen absorption may also be approximately calculated for cotton oil from its iodine number by multiplying this by .063 ($\frac{1.6}{2.54} = .063$).

For cotton oil the number thus obtained is 6.7 per cent.

ELAIDINE REACTION.

Oleic acid under the influence of nitrous acid is converted into an isomeric elaidic acid.

In like manner triolein $C_3H_5(OC_{18}H_{33}O)_3$ is converted into elaidine. This substance is formed in crystalline masses, and its melting-point is variously given at 32° to 38° . Following is the method of applying the elaidine test known as Pontet's process in the municipal laboratory of Paris :

	Grams.
Of the oil to be tested	10
Nitric acid	5
Mercury	1

Place in a test tube and shake vigorously for three minutes until the mercury is dissolved; allow to stand for twenty minutes, and shake again for one minute.

In from one to three hours the sample becomes hard. Olive, pea-nut, and lard oils give the hardest elaidines. Copper may be used instead

* Zeit. f. Angew. Chemie, June 1, 1888, p. 316.

† Moniteur Scientifique, Vol. 13, p. 263 *et seq.*

of mercury, in which case the nitric acid should be somewhat diluted. The red vapors produced by the action of iron on nitric acid may also be conducted directly into the oil.

One part of the strong nitric acid may also be shaken with three to five parts of the oil and a solution of nitrite of potash added drop by drop with constant shaking.

Attempts have been made to measure the relative hardness of the elaidine produced by the distance which a plunger carrying a known weight would sink into it, and the data thus obtained have been used for quantitative calculations.

SPECTROSCOPIC EXAMINATION.

The absorption spectrum of an oil depends upon the character of the coloring matter contained therein. Many vegetable oils give a spectrum characteristic of chlorophyll.

Cotton oil gives a banded absorption spectrum.

The use of the spectroscope in examinations for lard adulteration is probably not as extensive and general as the merits of the process would warrant.

FURTHER QUALITATIVE REACTIONS.

There are other qualitative reactions which might sometimes prove of value in the examination of lard and its adulterations.

These are the methods of Chateau, Fauré, Heydenreich, Penot, Crace Calvert, Flückiger, and Glæssner.

A full description of these methods is given by Benedikt.*

ABSTRACTS OF METHODS OF LARD ANALYSES, WITH RESULTS THEREOF.

[Employed in the case of *McGeoch, Everingham & Co. vs. Fowler Bros.*, before Chicago Board of Trade.]

Much progress has been made in the science of lard analysis since the famous case of *McGeoch, Everingham & Co. vs. Fowler Bros.*, the notes of which have been published in pamphlet form by Knight & Leonard, Chicago, 1883.

The complaint against the Fowler Bros. rested on the charge that they had sold prime steam lard which contained other than hog fat. The complaint was brought before the Chicago Board of Trade, and was heard by the board of directors thereof. Samples of the suspected lard were submitted to a large number of chemists, and an abstract of their methods of analyses and the results obtained follows :

TESTIMONY OF DR. P. B. ROSE.†

He can generally tell, when a sample of prime steam lard is sent to him, if there have been any impurities put into it, by examining its color and quality; the samples are sent to him for the purpose of seeing whether the lard is up to the proper

*Analyse der Fette und Wachsarten, p. 198, *et seq.*

† Pamphlet mentioned, p. 116.

standard, whether it is off-color, or anything of that sort; sometimes lard is of too dark a color; a small quantity of tallow in lard could not be detected by its appearance to the naked eye; a thousand or twelve hundred pounds of tallow put into one or two tanks could not be detected by the eye; he thinks during last November only a thousand or twelve hundred pounds of tallow was received into the house from all sources. Tallow fat is worth $7\frac{1}{2}$ to 8 cents per pound; he has never tried it, and does not know how much tallow could be put in a tank of lard without it being detected. 25 or 20 per cent. could be detected, and he thinks 15 per cent. could be readily detected by the naked eye and by the taste; he has never tried 10 per cent; he thinks an inspector would readily detect 15 per cent., and with 10 per cent. of tallow he thinks an inspector would discover there was something wrong.

TESTIMONY OF PROF. M. DELAFONTAINE.*

CHICAGO, June 6, 1883.

To whom it may concern :

This is to certify that on or about the 22d of May and the 2d of June, 1883, I received from Mr. Mixer, provision inspector of the Board of Trade, three samples of lard, respectively marked 1, 2, and 3. Mr. P. McGeoch requested me to analyze them, and I find that neither of them is pure hog fat. Samples Nos. 1 and 2 gave indications of cotton-seed oil, and both contain a percentage of beef stearine (or a corresponding quantity of beef tallow) exceeding 10 per cent. Owing to the smallness of the quantity of No. 3 at my disposal, I can not testify positively and beyond reasonable doubt about the presence or absence of cotton-seed oil, but the proportion of beef stearine is at least equal to that found in the other samples.

M. DELAFONTAINE.

The experiments were all comparative; the same weight of each substance and the same bulk of solvents used, drawn from the same supply; the vessels were of the same kind and capacity; the experiments were conducted on the same table, at the same window, etc.; nothing was different but the final results for different samples; temperature between 12 and 15 degrees centigrade. For the detection of cotton-seed oil olein was extracted, as usual, and tested by the elaidine test (the taste and smell were noted too). For the extra stearine the lard was treated with eight or nine times its weight of pure alcohol and ether, half and half, allowed to stand twenty-four hours, liquid then poured out and replaced by a little over half as much again of the solvent, shaken often, filtered after eight or ten hours, dried, weighed.

First. What quantity of lard did you operate upon?

Answer. For some experiments on about $4\frac{1}{2}$ grams; for others on twice that quantity; for others on 20 grams.

Second. What was the liquid you used to dissolve the lard in? if a mixture, state what proportions of each liquid.

Answer. Half Squibb's ether, and half Squibb's absolute alcohol.

* * * * *

Fifth. Did you heat the lard and add to it the mixture, or did you simply add the lard and then apply the heat?

Answer. The lard was heated to about 70 degrees C.

* * * * *

Seventh. What was the shape and size of the vessel in which you thus treated it?

Answer. Cylindrical glass jars, glass-stoppered, graduated, holding 50, 100, and 200cc.

Eighth. Did you decant the liquid off?

Answer. I did.

* * * * *

* Op. cit., pp. 139, 140, 141, 142, 143.

Tenth. After filtering or decanting as above, how did you treat it, or where did you keep it before weighing?

Answer. Dried it in an air-bath.

Eleventh. Did you weigh the residue while on a filter or in a beaker, or evaporating dish, or how?

Answer. On the filter.

Twelfth. What was the exact weight of the residue found?

Answer. I have kept a record only of the results; the only figures that I can find just now of the actual weight of the residue are the following:

4.25 grams of lard, No. 3, gave 275 milligrams.

4.7 grams of pure lard gave 350 milligrams.

19.74 grams of pure lard gave 200 milligrams.

9.4 grams of lard, No. 3, gave 600 milligrams.

In making his sample test of pure lard he took his material chiefly from the leaf lard and from the sides of the hog; some of it was salted and some was fresh; before rendering it he cut the material into very small pieces, and allowed it to stand in a large volume of cold water for some time to take the salt out; it was then filtered out in pans and rendered on a sand-bath, that is, pans full of sand and heated from below, so as to get an even temperature and not burn the lard; after rendering, the lard was filtered, in order to remove any tissue or foreign matter from it; he is quite sure he got, into 1 per cent., all the lard there was in the material; the temperature at which the lard was rendered was 175 to 200 degrees centigrade,* which is much higher than is necessary to break up the cells and melt all the stearine there may be in the lard. In getting the samples of lard from a packing-house he asked for pure prime steam lard; in testing that sample he found it to run a little higher in stearine than the lard he rendered himself; he can not, of course, say that the sample procured from the packing-house was perfectly pure, because he did not himself see it rendered. The solvent he used was absolute alcohol and the strongest of Squibb's ether; he always measured the solvent; the melted lard was at about 70 or 75 degrees centigrade, when the solvent was applied, so as to be sure the palmitine would remain in solution. After the solvent was mixed with the lard he did not ascertain its temperature; he had no use for that; the lard was allowed to stand in the solution before decanting about fifteen to eighteen and sometimes twenty-four hours, during which time it was kept in cold water, at a temperature 12 to 15 degrees centigrade, always below 15 and sometimes a little lower than 12; he does not know what the temperature of the room was in which the mixture was allowed to remain. The mixture of alcohol and ether, after being added to the lard, was well shaken; after the first solvent had been decanted, he replaced it with about half as much of fresh solvent as had been first used, shook it well and often for two or three hours, and then allowed it to stand ten or twelve hours or so, sometimes over night, again shook it several times, then filtered, and in order to avoid an error that might arise from the liquid evaporating and leaving a part of the residue too hard, he pressed it between blotting papers so as to absorb all that was not properly residue, then dried in an oven and weighed it; sometimes, while it was on the filter, he poured more of the solvent on it and again filtered so as to dispose of all that was soluble. He can not see what the residue could contain except stearine, unless it might be a small quantity of palmitine; he tested the residue by determining its melting point and its solubility, and that showed it to be stearine; there is no difference in the chemical characteristics of the pure stearine procured from the fat of beef, mutton, or pork; they are the same thing as far as he knows, and he does not know of any difference in the chemical reaction of these different kinds of stearine; he does not attempt to distinguish between them; he does not know certainly how much pure stearine lard actually contains; it varies; he has never found it to exceed 2 per cent. in pure lard, and sometimes it runs as low as three-quarters of 1 per cent. when subjected to the

* Probably Fahrenheit is meant.

process for extracting it he has described; very likely it would vary that much in lard made from different parts of the same hog: he speaks on these points from his own experiments; he has not looked for authorities on this subject; he is now performing some experiments which he hopes will throw some light on this branch of the subject.

* * * * *

In testing for cotton-seed oil he extracted the olein by means of absolute alcohol, heated, allowing the liquid to cool and then filtering and drying off the alcohol; he takes a glass flask or anything capable of holding the lard and pours over it some absolute alcohol, and boils the two together for a few minutes, then allows the mixture to cool; this produces a crystallization; and then having kept it cold for a number of hours he filters it, and the liquid is for all practical purposes a solution of olein and alcohol; the alcohol is then driven off from it and what is left is olein. In the case of these lard samples he treated the olein by the elaidine test, using as a liquid sulphuric acid saturated with the red fumes of hyponitric acid; by this treatment the olein of oils is turned into a hard solid mass; olein is naturally a liquid, but when the test is applied to cotton-seed oil the oil remains floating. The same test applied to pure lard oil or pure olive oil soon turns the oil hard. If cotton-seed oil and lard oil are mixed with this liquid the mixture will solidify only after a much longer time than would be required to solidify pure lard oil, or often it will not solidify at all, depending upon the proportion of the cotton-seed oil; he took a glass test-tube and put into it a certain quantity of the olein to be tested, and the acid to about half the bulk of the olein, shook it well, and kept the tube at a temperature of about 10 degrees centigrade; he observed the time it took for the liquor to solidify. Nitric acid for use in the elaidine test is not reliable and he did not use it; he depends upon the absence of solidification after half an hour to determine the basis of reaction. Lard oil solidifies pretty quickly when treated by the acid he employed; cotton-seed oil does not for several hours.

He does not know of any writer who has stated that cotton-seed oil can not be detected when it is present in less proportion than 5 to 10 per cent.; he has that information from personal conversation with others. His method of analyzing lard is not one published in the books, as far as he knows; he adopts it mainly as the result of putting this and that together. He is not willing to take ten samples of lard prepared by a competent and reliable expert, whose certificate as to what they contain shall be placed in the hands of the president of the Board of Trade and stake his reputation on being able to tell which are adulterated and which pure, using in the analysis the methods he has employed in testing the samples in respect to which he has been testifying, because a mixture can be made with fats or some foreign oil which he has not sufficiently studied to be able to certainly detect such substances; his examinations have been with reference to detecting substances which are most likely to be used for the purpose of adulterations, such as tallow and some other substances. In the case of a mixture of equal proportions of pure lard with a lard from which, say, half of the lard oil has been expressed, leaving the mixture deficient in lard oil to the extent of 25 per cent., that mixture would be found to contain more stearine than pure lard.

EVIDENCE OF WILLIAM HOSKINS.*

CHICAGO, June 5, 1883.

This certifies that I have analyzed a sample of lard received from Mr. C. H. S. Mixer, marked No. 3, and find that it is adulterated with at least 20 per cent. of beef stearine or its equivalent of tallow; and further, I find evidences of the presence of cottonseed oil, or one of its derivatives.

G. A. MARINER.
Per HOSKINS.

* Op. cit., pp. 147, 148.

CHICAGO, June 1, 1883.

This certifies that I have analyzed two samples of lard, marked respectively No. 1 and No. 2, received from Mr. Mixer on May 30, 1883, and find that both are adulterated with beef stearine or its equivalent amount of tallow to the extent of at least 20 per cent.

G. A. MARINER.
Per HOSKINS.

One of his methods was by taking equal proportions of alcohol and ether, and into that mixture putting a certain amount of pure lard, and also an equal weight of the samples to be tested. The lards were warmed, and were then poured into the vessels containing the mixture under exactly the same conditions. After some time more or less of the stearine separated. In pure lard, treated in the way he has described, the separated substance, which is chiefly stearine, rarely exceeds 1 per cent. One of the samples given him by Mr. Mixer gave less than 4 per cent.; one gave over 5 per cent. Another process, known as Blythe's pattern process, is to take a piece of glass, chemically cleaned, and having a thin film of water on it. On this is dropped a drop of the melted substance. In the case of lard one pattern is produced, in the case of tallow a different pattern, and in the case of the mixture of the two a still different and intermediate pattern is produced. He regards this as an absolute test and one easily applied. Another test used is to ascertain the difference in time taken in saponifying samples. Tallow takes much less time to saponify than lard does, under proper and the same conditions. This process gives quite accurate results. These are the chief tests he depended upon in his chemical examinations of the samples now in question. The processes he has described are recognized by authorities, and have all been published as authority. He has during the past two winters had considerable experience in examinations as to the adulteration of butter, and has studied the subject of fats to a considerable extent. He considers the results of his examination of the samples given him by Mr. Mixer as conclusive in respect to their quality; there is no possibility of a doubt as to the correctness of his conclusions in respect to them. In regard to the presence of foreign oil in lard there is an absolute test, known as the elaidine test. It especially applies to drying oils. Nitrous oxide is made by heating mercury with nitric acid. In treating non-drying oils with it the point noted is the fat which is solid. It makes no such combination with drying oils, and when they are in large quantity they separate and come to the top and can be seen as liquid. In other cases they form more or less of a semi-solid; he thinks almost anybody could see the difference when pointed out, although it requires considerable experience in the use of the microscope to be able to detect these differences unaided. All his examinations were carried on parallel with examinations of samples of pure lard rendered by himself, at least a part of which was leaf lard. He thinks there may be a little difference between the proportions of stearine in leaf lard and fat taken from the sides of the hog. It is, however, but slight. After all his examinations of the samples in question in this case, it is his absolute conclusion that the lard is adulterated to the extent of 20 per cent. of foreign material. He calculates the percentage of adulteration from the basis that, as pure lard never contains more than 1 per cent. of pure stearine, and that tallow contains about 9 per cent., therefore, as these samples contained none less than 4 per cent., there must have been added to the lard beef tallow, or some of its derivatives, sufficient to account for the 3 per cent. of excess, and as three nines are twenty-seven, he concludes there was over 20 per cent. of adulteration in the samples. The test of adulteration is by the amount of pure stearine found in the sample.

EXACT METHOD OF ANALYSIS.*

A portion of the fat was warmed and mixed with about ten times its weight of a mixture of equal parts of absolute alcohol and ether. After allowing it to stand about twenty-four hours the residue was filtered and weighed.

*Op. cit., p. 149.

A plate of chemically clean glass was covered with a film of water, a drop of the melted fat was dropped upon the plate, and the patterns noted.

A portion of the fat was saponified with an equal weight of sodic hydrate dissolved in water, and the time occupied in saponifying noted.

A portion of the fat was treated with a solution of hyponitric acid and sulphuric acid, and the time necessary for the solidification of the elaidine noted.

In all the above parallel experiments were made under exactly the same circumstances in every respect.

W. HOSKINS.

The above methods rests chiefly on the percentage of insoluble residue after treating the fat with a mixture of ether and alcohol as described.

METHOD OF J. M. HIRSH.*

I melt the sample and dissolve it in purified naphtha, leaving it there at rest at a temperature of about 70° F. for twelve hours, when added stearine or tallow will deposit, while pure lard will show no deposit, or barely a trace. The amount of the deposit increases considerably in the next twelve hours in a mixture with stearine, but little in pure lard. The test being made in a graduated tube, the proportions can be read off without possible error by washing, weighing, or measuring. After the time mentioned the solids of the lard deposit. The remaining solution I treat with nitric acid, which renders crystalline the animal oil (producing elaidine), but leaves the cotton-seed oil a colored liquid.

As a rule the melted sample has minute fibers of cotton floating when it is contaminated with cotton-seed oil; this test is simple and infallible; for this reason I omit to mention other corroborative tests.

J. M. HIRSH.

FURTHER REMARKS BY J. M. HIRSH.†

Lard will give a reaction of elaidine as well as cotton-seed oil, but a time amply sufficient to make elaidine from lard or any animal oil must be greatly exceeded to get the same result from cotton-seed oil. If linsed oil or cotton-seed oil, or the two mixed, are boiled for five minutes with a fume of nitric acid, there will be no apparent change except that they become colorless; they will have to boil an hour or two before separation takes place; in half an hour or so they would become a solid, like stearine. Animal oils treated in the same manner will be solidified and converted into elaidine in five minutes. In applying the elaidine test he first took all the crystals out of the solution, then drew off from the tube all the olein and the benzine, put the nitric acid into that liquid, and heated it; the benzine evaporated quickly; the heating was continued for a few minutes longer, and then it was allowed to cool; the crystallized deposit of elaidine he considered as from animal oils, and what was left, after withdrawing the liquid, he considered was from an admixture of some other oil. In a lard rendered at a high pressure of steam there would be a greater amount of stearine than in one rendered at a low pressure, the original material being the same. If lard is rendered and run into a large reservoir holding, say, 250 tierces, and there allowed to stand for a time, the lard from the bottom of that reservoir would contain a greater amount of stearine than would that drawn from the top. There would be a great deal of difference. The heaviest lard would settle in the reservoir. The difference in the stearine would not be more than, if so much as, 2 to 4 per cent. of chemically pure stearine. A good deal would depend on the depth of the tank or reservoir and on the temperature maintained in the lard. If, thinks in lard drawn from the top or bottom of such a reservoir there would not be as much difference in the stearine as he has found in the lard delivered him by

* Op. cit., p. 154.

† Op. cit., p. 155.

Professor Delafontaine over what pure lard should contain. There is some difference in lard on account of the season at which the hogs are killed, and on account of the age, feed, and other conditions of the animals from which the lard is made.

METHOD OF PROF. C. B. GIBSON.*

Five cubic centimeters of the molten sample, at a temperature of 90° to 100° C., were dissolved into 45cc of half solution of absolute alcohol and ether; this mixture was allowed to stand eight hours, at a temperature of 5° to 10° C., and the stearine allowed to crystallize out; the supernatant liquid was then poured off and 25cc of the fresh solution added to dissolve any remaining olein and palmitin, and allowed to stand twelve hours at 5° to 10° C. The liquid was then filtered off and the residue collected on a tared filter; this was washed until no more fat globules were deposited on evaporating a drop of the washings (alcohol and ether solution); the filter and contents were then dried at a temperature of 30° to 40° C., and by means of desiccator, weighed and the result calculated. All samples were treated at the same time and under the same conditions. In the test for cottonseed oil, I submitted 9cc of the molten lard to the action of sulphuric acid, saturated with nitrous and nitric anhydride, 7cc; the test kept at a temperature of 5° C., until solidification took place, which, in the case of the presence of cotton-seed oil, is produced only after long time; from these results I drew my conclusions. All samples were tested at same time and under same conditions.

C. B. GIBSON.

REMARKS BY PROF. C. B. GIBSON.†

He took the two samples that he received from Professor Delafontaine, a sample he prepared himself, and a sample he procured in the market, said to be absolutely pure lard, and treated them all by the methods he has described in his written statement; all the samples were treated in corked tubes; the samples all produced different results, some considerably different, and in the two samples he rendered himself there was a slight variation; he tested the process by comparison with the pure lard he had rendered; he did not make up any samples of mixtures; in rendering the pure lard for standard samples he cut the fat very fine and put it into a large porcelain dish, and adding water, boiled it from forty-five minutes to an hour; then he squeezed out a portion of the fat, and subjected the residue to a little greater heat and extracted all the fat he could possibly get out by any ordinary squeezing method; the lard then contained some water, which he removed as far as he could by decanting; then heated the lard over a sand-bath, being careful not to heat it so much as to burn it; but he certainly had it at a sufficiently high temperature to hold the greater part of the stearine in a molten state, and pass it through the filter.

It would probably depend a little on circumstances which of two samples of lard, one rendered at a low pressure and the other at a high pressure, would contain the most stearine; speaking casually he should say the one rendered at a high pressure would contain the most; he means by high or low pressure, a greater or less pressure in squeezing out the lard as is ordinarily done in a small way; he should think there might be a tolerable variation in the quantity of lard stearine from this cause, but of the pure stearine there ought not to be such a remarkable difference. It depends entirely upon when the lard is produced, when the hog is raised, when killed, what fed upon, and perhaps other conditions, as to how much stearine there may be in lard; authorities differ on the subject; some claim there is as high as 33 per cent., others less, of lard stearine; as far as he has been able to learn, lard stearine varies from 30 to 40 per cent., and pure or chemically pure stearine from less than 1 per cent to about 3 or $3\frac{1}{2}$ per cent. depending upon the conditions he has referred to; his personal examinations have shown a variation of from a little under 1 per cent. up to about $2\frac{1}{2}$ per cent. He should think, the better the hog is, the better would be the

* Op. cit., p. 157.

† Op. cit., p. 158.

fat from it, and probably the richer the fat in stearine, and this would apply to the lard rendered from a large number of hogs of average fine quality, as compared with the lard rendered from a large number of average inferior quality; samples drawn from different parts of a large tank holding 250 ierces might vary a little, depending upon whether the lard put into it was thoroughly mixed, at what temperature or how fast it had been cooled, and other conditions.

TESTIMONY OF C. GILBERT WHEELER.*

In treating the lard the particular process upon which he relies—although he used others, some of which pointed to the same conclusions and others to no special result—is based upon the insolubility of pure stearic acid in a mixture of absolute alcohol and ether. In treating by that method he takes a given amount of lard and nine times as much of a mixture, composed of equal parts of absolute alcohol and ether, places them in a closed vessel, with a graduated scale upon it, agitates, and exposes to a low temperature, the agitation being repeated a few times; then, after standing for about twelve hours, the supernatant liquid is poured off and as much more of a fresh supply is added, and again shaken; after standing again for about twelve hours the liquid is entirely poured off, the residue collected, dried and weighed, and its amount compared with that obtained in the same way from both pure lard and impure lard. Pure lard should give a certain per cent. of residue, impure lard gives more; the residue so obtained is pure stearine. In the case of pure lard rendered by himself for a standard of comparison in his investigations of the samples in question, the amount of residue obtained was nine-tenths of 1 per cent. In the samples he was to examine, No. 1 gave 3.6 per cent., No. 2 gave 3.29 per cent., No. 3, gave 2.75 per cent. The process was conducted at a temperature of about 75 degrees Fahrenheit; the amounts of lard taken were, for the pure lard, 10 grams; of sample No. 1, 10 grams; No. 2, 5 grams, and No. 3, 5 grams.

The evidence of which an abstract has been given was on the side of the prosecution and the charge of adulteration appears to be well founded in the light of the evidence given. Following is a brief abstract of the chemical evidence introduced by the defense:

TESTIMONY OF DR. ROBERT TILLEY.†

Dr. Tilley made a microscopical examination of the samples *in lite* and gave the following certificate:

CHICAGO, June 29, 1883.

This is to certify that on the 9th day of June, 1883, I received from Prof. W. S. Haines samples of lard labeled, respectively, No. 1 Fowler, No. 2 Fowler, No. 3 Fowler; that I have examined the same microscopically, and that I can find no evidence of adulteration; and consequently, in the absence of such evidence, I believe said samples to be pure lard.

ROBERT TILLEY, M. D.

Dr. Tilley added the following explanatory remarks:‡

He has examined fats when crystallized in the manner described by Mr. Hoskins; he has never had a sample of pure palmitine to examine, and therefore can not say whether there is any difference between the crystals of it and the crystals of stearine; he can not say whether the appearance of the crystals, in the methods used by himself, depends upon the relative proportions of stearine and palmitine, because he does not know of any means, apart from temperature and pressure, of thoroughly isolating palmitine and he has never done it; he is of the opinion that the greater part of the solid fat of either the beef or the hog is stearine, and not palmitine; the constit-

* Op. cit., p. 160.

† Op. cit., pp. 165, 166.

‡ Op. cit., pp. 167, 168.

nent parts of lard are oleine, palmitine and stearine; the crystals of stearine seem to be modified according as the substance is mixed with beef stearine, or hog stearine; his answers are in part from the books and in part from his own experience; he has never had the opportunity of observing any modifications in the crystals by an increase or diminution of the palmitine in a specimen, and consequently can not say whether or not the crystals would be modified as stearine or palmitine predominated. In applying the method of microscopical examination to lard, described by Mr. Hoskins, he takes a microscopical slide and cleans it, chemically, then puts on the slide a small quantity of the specimen to be examined, puts it in a water-bath, at the temperature of boiling water, covers it with a covering glass and allows it to cool as slowly as possible; he has made a number of experiments of this kind, sufficient to satisfy him that he could find no distinction. The method he used, and upon which his conclusions as to the lard in question in this case are based, was as follows: He dissolved the specimens in sulphuric ether, allowed the ether to partially and slowly evaporate and crystals to deposit, then decanted the remainder of the ether, washed with ether and decanted again, then treated with absolute alcohol; after which the crystals were examined under the microscope.

* * * * *

Commercial stearine may or may not include palmitine, according as heat and pressure are used in the separation; he thinks he understands the manufacture of commercial stearine; it would depend upon the temperature to which it is subjected whether it would contain all the original parts of the fat, except what oleine may have been pressed out; palmitine is said to liquify at a temperature of 45° C. consequently if the fat material is subjected, at that temperature, to a pressure similar to that used for extracting the oleine, the palmitine would also be forced out; he does not know, as commercial stearine is usually made, that it is ever subjected to a temperature high enough to press out the palmitine, but he has seen it subjected to a temperature in the manufacture of candles—obtaining stearic acid—when it certainly would be pressed out; as to whether the crystals produced from commercial stearine would be the same as those produced from what has been called by other witnesses chemical stearine, he can only say that he is not acquainted with the peculiar characteristic features of palmitic crystals; the size and general character of the crystals depends on the temperature and slowness of evaporation; there is a difference between the crystals of pure stearine from lard and that from tallow; he states this upon the theory that the stearine he has obtained was pure stearine, but inasmuch as he is not sure that the stearine he obtained did not contain a mixture of palmitine, he desires to make that qualification. As between the crystals obtained by his process and those that caused the so called grain of the lard, he could not, chemically, see any distinction whatever; microscopically this grain seems to be the crystal, in a form very much resembling an ordinary roadside bur, when examined under the microscope, but the details are so ill-defined that it is simply impossible to make any differentiation; the crystal itself would be a solution, if melted. He does not think crystals obtained from lard itself, without extracting the oleine, would be as valuable as those from the stearine, because it is acknowledged that that there is at least 60 per cent. of oleine in the lard, and that 60 per cent. of extraneous matter would, he thinks, necessarily render the crystals more difficult to differentiate than if crystallized from the stearine alone; probably the crystals which characterize the grain of the lard are the same as those obtained by the method described by Mr. Hoskins; they might be the same thing, but yet so modified by the influence of the oleine, that the peculiarities of the crystals would be less prominent; they would be more or less stunted, or their development favored. As to whether or not the method requiring the least manipulation would be likely to give the best results would depend entirely upon the advantages gained by the manipulation, and the care with which it is done. In transferring the crystals to the microscope, he takes a small glass tube—a pipette—cutting off for each observation, with a file, the piece used in the previous case, so that it is perfectly clean as it goes into the liquid;

it is difficult to get the crystals under the microscope in perfect form; but he thinks the Board will have an opportunity to see how perfectly they can be gotten out, by an exhibition of photographs of those they have used for this examination; it is necessary that the crystals be washed in order to obtain a plain, clear-cut specimen.

CERTIFICATE OF MICROSCOPIC EXAMINATION BY DR. W. T. BELFIELD.*

CHICAGO, June 29, 1883.

On June 12, 1883, I received from Prof. W. S. Haines three samples marked "Fowler 1," "Fowler 2," "Fowler 3," respectively. I have submitted these samples to microscopical examination for the purpose of detecting the presence of beef tallow. By either one of two methods I have satisfied myself of my ability to detect the presence of beef tallow in lard, whenever the admixture contains 10 per cent. or more, by weight, of the tallow.

By neither of these methods did I detect the presence of tallow in the samples above mentioned; I am therefore convinced that these samples do not contain an amount of tallow equal to 10 per cent. of the weight.

I have as yet no knowledge of any methods of microscopical examination whereby I can detect an admixture of less than 10 per cent. of tallow *with certainty*, but I have never obtained in the samples above mentioned any appearances other than those which may be presented by pure lard.

WILLIAM T. BELFIELD.

Dr. Belfield further says:†

He is not familiar with the manner of manufacturing prime steam lard; he supposes the tanks are covered during the process of rendering and that when put into tierces it is covered. If he found in a sample of lard that had been kept covered an excess of cotton fibers of special characteristics he should not draw any inference as to how they came to be there; if lard in which cotton fibers were found was chemically tested for the determination of the presence of cotton seed oil, and the chemical test was supposed to detect it, he would not be inclined to attach any more value to the chemical test on account of finding individual cotton fibers in the lard by a microscopical examination. He does not wish to be understood as saying no one can detect cottonseed oil in lard by microscopical examination; he meant to say he could not do it. He has not examined the crystals from isolated stearine or isolated palmitine. In crystallizing stearine and palmitine, in the manner described by Mr. Hoskins, he should think the appearance of the crystals would depend on the relative proportions of each, but as he has never worked isolated palmitine he can not say certainly. In the method he uses the appearance of the crystals does not depend on the relative proportions of the stearine and palmitine in the mixture on which he operates; there is said to be a difference between the crystals of stearine and palmitine. He ordinarily transfers crystals from the liquid to the microscopic slide by means of a clean pipette; it can be done, and he has done it, by means of a clean knife blade, or something of that sort. The objection to Mr. Hoskins's method is that the characteristic crystals of lard and tallow are not formed by it; there is so much granular matter that it gives crystals so nearly alike that he can not differentiate between those of lard and of tallow; a principle to be worth anything, in matters of this kind, must detect minute adulterations. The question of discovering adulteration in lard by microscopical examinations is a new one, at least it is so to him. New discoveries of facts by microscopy, which may be subsequently well established, are sometimes questioned even by experienced microscopists, if they fail to follow the methods and directions of the discoverer, as has been the case in some rotatable instances. These persons may attempt to follow the discoverer, but, doing so imperfectly, fail to secure the expected results. This fact, however, does not apply to his judgment on the method pursued by Mr. Hoskins, because he followed Mr. Hoskins's method as he described it.

* Op. cit., p. 176.

† Op. cit., p. 177.

CERTIFICATE OF PROF. PLYMMON S. HAYES.*

CHICAGO, *June 29, 1883.*

On the 9th day of June, 1883, I received three samples of lard from Prof. W. S. Haines, marked, respectively "No. 1 Fowler," "No. 2 Fowler," and "No. 3 Fowler." These samples of lard I examined microscopically, after having crystallized the stearine found in the samples from solution, and was not able to detect any beef stearine whatever.

PLYMMON S. HAYES.

Professor Hayes adds the following observations:†

He has examined crystals which he obtained by dissolving the stearine of beef tallow, of mutton tallow, and of lard—three samples in absolute alcohol; the substances were dissolved and crystallized, redissolved—and recrystallized, and finally the alcohol was evaporated off; he considered those the crystals of pure stearine; he has never examined the crystals of pure palmitine to his knowledge; he does not know what, if any, difference there may be between the crystals from pure stearine and pure palmitine; he does not think the appearance of the crystals obtained by himself depend entirely upon the relative proportions of stearine and palmitin in the specimens; the crystals from a fat, consisting of oleine, stearine, and palmitine, which has been warmed and then slowly cooled, would be those of stearine and palmitine, probably more or less modified, as one or the other was in excess, and also by the presence of the oleine. Molten lard is a solution of oleine, stearine, and palmitine. When this is allowed to cool slowly and crystallize the crystals are valueless, for the reason that they give no distinctive difference when examined plainly or by means of the polariscope. He has examined crystals in the manner described by Mr. Hoskins; he will not say Mr. Hoskins's method is wrong, either in principle or in application, but it does not produce results that are plainly marked. He pursued that process for over two weeks before he abandoned it as useless. In his examinations by that method he took the sample to be examined and put it on a microscopic slide, put a cover-glass over it, and applied heat until it was thoroughly melted, and then set it aside to cool; sometimes he allowed it to cool very slowly, and sometimes he cooled it by means of a cooling apparatus; in neither case did he get satisfactory crystals; he never tried cooling it by means of a hot iron allowed to cool slowly with the lard.

CERTIFICATE OF DR. I. N. DANFORTH.‡

CHICAGO, *June 29, 1883.*

I hereby certify that on the 8th day of June, 1883, I received from the hands of Prof. Walter S. Haines, of Chicago, three specimens of lard, numbered 1, 2, and 3, respectively, and said to have been manufactured by the "Anglo-American Packing and Provision Company," of Chicago; that I was requested by said Haines to examine said specimens microscopically, and state my opinion as to their purity or impurity; that I have examined said specimens as thoroughly and carefully as the time allowed would permit, and am of opinion that they are composed entirely of the fat of the hog.

ISAAC N. DANFORTH.

Dr. Danforth in explanation said: §

He has examined the crystals of pure stearine; he procured the first specimen from Professor Haines, and afterwards prepared specimens by methods used by chemists; he has not examined the crystals of pure palmitine; he does not understand that any

* Op. cit., p. 179.

‡ Op. cit., p. 182.

† Op. cit., pp. 179, 180.

§ Op. cit., p. 184.

method has been devised for absolutely isolating palmitine, and he does not know whether or not there is any difference between the crystals of pure stearine and those of pure palmitine; he can not say whether the crystals procured by him depend on the relative proportions of stearine and palmitine in the substance from which they are procured; while he does not claim to speak as authority on chemical subjects, he thinks the substance from which these crystals were obtained was particularly pure stearine; he can not positively say whether the crystals obtained from a fat consisting of oleine, stearine, and palmitine would be those of stearine and palmitine more or less modified as one or the other was in excess; he thinks the presence of the oleine would modify the crystals somewhat, but to what extent he is not able to say.

METHODS FOLLOWED IN MAKING MICROSCOPIC INVESTIGATION BY MESSRS. TILLEY, BELFIELD, DANFORTH, HAYES, AND ROSE.*

Dr. Tilley's formula.—For the production of the crystals of stearine, whether from beef or hog products, I dissolve the fat in question in sulphuric ether, 10 grains in 2 drachms; allow the ether to partially and slowly evaporate, and consequently crystals to deposit; decant the ether remaining, wash with ether, decant again and treat with absolute alcohol, then examine the crystals under microscope.—Robert Tilley, M. D.

Dr. Belfield's formula.—Ten grains of the fat are dissolved in Squibb's ether; the quantity of the latter may be 1 drachm or 2 drachms, or instead of the ether, a mixture of this substance with absolute alcohol in equal parts may be employed as a solvent. The solution is allowed to stand in a test tube uncorked for twenty-four hours, at ordinary temperature; at the expiration of this time crystals are observed at the bottom of the tube; these may be examined directly through the microscope, or supernatant liquid can be poured off and replaced by a drachm of absolute alcohol; this is subsequently removed and the crystals examined; the crystals may be mounted for examination in the solvent used or prepared dry. When specimens of pure lard, pure tallow, and mixture of both, within certain proportions, are treated in this way, characteristic crystals are formed by means of which the identity of the specimen can be established. Essentially the same results are obtained when the method is varied by changing the quantity of the solvent, within certain limits, or by repeated washings with alcohol.—William T. Belfield.

Dr. Danforth's formula.—The oleine is first extracted from the specimen to be examined by the use of absolute alcohol or a mixture of alcohol and ether in equal parts, or by any other method the experimenter may choose to adopt; the remaining stearine is then dissolved in any one of a number of menstrua, as, for example, ether, turpentine, benzole, oil of Scotch pine, or any other solvent of stearine; I usually employ turpentine; from this solution crystals are allowed to form, and the resulting crystals are then mounted upon glass slips prepared for the purpose in Canada balsam diluted with twenty-five per cent. chloroform, or a solution of damar, or they may be examined in the original solvent. Another method I have used recently to a considerable extent, is to place the specimen to be examined immediately in some solvent of stearine, as ether, or benzole, or turpentine (usually the latter), in the proportion of ten grains of the specimen to be examined to a fluid drachm of the solvent, without first extracting the oleine; I then watch carefully for the formation of the first crop of crystals; these crystals are then immediately examined after being mounted in either one of the media that I have already mentioned. Recently I have employed a solution of balsam in chloroform as a mounting medium because it gives the clearest field. The crystals are examined by the use of a one-quarter inch objective and an "A" eye-piece, giving a magnifying power of about two hundred and fifty to two hundred and sixty diameters. The difference in appearance between the lard stearine and beef stearine crystals is clear and definite.—Isaac N. Danforth.

* Op. cit., pp. 197, 200, 201.

Professor Hayes's formula.—Take a suitable vessel that can be well corked and put in it some of Squibb's ether, add to it lard, or preferably the stearine from the lard in question, until at a temperature of 85° F. A slight portion of the lard or stearine remains undissolved (the stearine may be obtained from lard by making a hot saturated solution of lard in a mixture of equal parts of absolute alcohol and ether, or some other solvent, and crystallize out the stearine by cooling the solution); the vessel is then very tightly corked and the ether heated until the solution is perfectly clear; then the vessel is placed in water, or left in a room, at a temperature of from 60° to 70° F.; the slower the solution cools the larger will be the crystals; the ether is then decanted from the crystals and the crystals washed with cold ether or a mixture of absolute alcohol and ether; the crystals are again washed with absolute alcohol, after which a fresh portion of absolute alcohol is put on the crystals and allowed to stand. The crystals may be mounted either in Canada balsam, glycerine jelly, or dry. In examining the crystals I usually employ a fifth objective and a "B" eye-piece. The difference in form of the beef and lard stearine crystals furnishes a ready means for their detection.—Plymmon S. Hayes.

Dr. Rose's formula.—Take 1 gram of the fat and dissolve in absolute alcohol, allow it to stand until crystals form, decant off the solution, and wash with repeated portions of absolute alcohol, finally remove the alcohol by heat, and dissolve in turpentine and set aside to crystallize; when crystals form remove with a pipette and examine them with the aid of a microscope; the crystals formed from beef products are quite different from those obtained from hog products.—P. B. Rose.

AN OUTLINE OF DR. ROSE'S METHOD OF CHEMICAL ANALYSIS.*

Take a definite quantity of the article to be examined, add nine times its weight of a mixture of equal parts of ether and absolute alcohol (the absolute alcohol used was but 93 per cent. alcohol), thoroughly agitate, place the jar in water, kept at 65° F. for twenty-four hours; the clear portion is then decanted off and about one-half as much more of the alcohol and ether is again added; agitate and allow the jar to stand for twenty-four hours in water at the constant temperature of 65° F.; the liquid portion should then be decanted off and finally filtered; the filter containing the residue placed in a jacketed filter filled with water and kept at the temperature of 65° F.; the residue on the filter is washed with a mixture of alcohol and ether, cooled to the temperature of 65° F.; the filter and residue are dried and weighed, the filter being counterpoised by a second filter.

The following are the amounts taken and the results:

	Grams.
Prime steam lard taken	10.905
Residue obtained	2.846
Kettle-rendered leaf lard taken	10.351
Residue obtained	3.250
No. 1 sample taken	10.065
Residue obtained	2.550
No. 2 sample taken	9.979
Residue obtained	2.524
No. 3 sample taken	9.599
Residue obtained	2.409

The other experiments were conducted in like manner except that the temperature was kept at 50 and 60° F., respectively.

P. B. ROSE.

*Op. cit., p. 202.

CERTIFICATE OF WALTER S. HAINES.*

CHICAGO, July 2, 1883.

This is to certify that I have examined the specimens of lard delivered to me by Col. Ezra Taylor and numbered 1, 2, and 3, and also the specimens placed in my hands by Dr. Robert Tilley, numbered 1, 2, and 3, and that I find them pure lard and unadulterated with tallow or cottonseed oil.

WALTER S. HAINES.

METHOD EMPLOYED BY PROFESSOR HAINES.†

To test for the presence of cottonseed oil I used the color test with strong sulphuric acid: from 5 to 10 grains of the lard are stirred up with one or two drops of the acid and the color produced is noted. Pure lard thus treated gives a color ranging from salmon to slate, cottonseed oil a dark olive brown, while lard mixed with cottonseed oil produces a well-marked mixture of the two colors.

To test for the presence of tallow I make a solution of lard in some slightly warmed solvent, such as turpentine, ether, or ether and alcohol; after a number of hours the crystals thrown down are examined (preferably after washing with a little absolute alcohol, or ether and alcohol) by the aid of a microscope; the form of crystals indicates whether the lard has been adulterated or not.

WALTER S. HAINES.

In explanation of his results, Professor Haines said:‡

In saying that he used essentially Professor Delafontaine's process, he meant that he had endeavored to follow the spirit of that process as he understood it. Professor Delafontaine insists that his examinations of the lard were all conducted comparatively with samples of known purity; and as his own were conducted in that way, it makes but little difference what temperature is employed; he always used the same temperature for the examination of the samples in question. He thinks Professor Delafontaine's process is not sufficiently accurately given to admit of being followed with entire precision; he does not now remember the temperature Professor Delafontaine used; he read that gentleman's certificate and evidence in this case in respect to his process. He (Prof. D.) states* that any chemist, on reading his certificate, will be able to tell his process. The certificate does not allude to temperature, and he concludes that, as nothing was said in it about the temperature employed, it was considered by Professor Delafontaine as of minor importance, or no importance; it is true, on cross-examination, Professor Delafontaine did mention the temperature, but he concludes that when a chemist presents with deliberation, in writing, an account of his process, and states, in connection with the certificate, that it is amply sufficient for any chemist to follow, and has excluded any mention of temperature in the certificate, the party attaches to temperature a very trivial importance, or no importance at all; it might have been considered an accidental omission, but for the fact that it was insisted that he should give all essential particulars of his process, and after the certificate was submitted, he, again and again, insisted that everything essential was stated in the certificate; and in view of these facts he (the witness) can not consider that the omission was by an oversight, especially as when, on the cross-examination, the temperature was developed it was not then spoken of as being essential. In all his experiments of treating the lard under consideration, concerning which he has testified, he either did the work himself, or saw it done from beginning to end; those performed in his own laboratory were conducted entirely by himself, in person, and whenever he left the laboratory, while the process was going on, the room was securely locked.

* P. 219.

† Op. cit., pp. 219, 220.

‡ Op. cit., pp. 220, 223.

In a mixture of 90 per cent. of lard and 10 per cent. of cottonseed oil, treated with sulphuric acid, as he has described, the color produced will be the salmon, with a tinge of slate peculiar to the lard, and in addition a tinge of the olive brown peculiar to the cottonseed oil; the shades of color produced by this process will probably be somewhat differently classed by different individuals, even as a result of seeing identical colors or shades of color; some might call what he describes as olive brown, a mahogany brown, and so of other tinges of color, depending upon peculiarities of vision in different individuals; some persons are slightly color-blind, others greatly so; he speaks of these colors as they appear to his own vision; if a person has not an eye trained to distinguishing colors, he might not, perhaps, discover the difference in these shades; the gentlemen who have been associated with him in these examinations have been unanimous in picking out the colors peculiar to the lard, and to the cottonseed oil, and became trained in that respect before pronouncing on the several samples; he has never applied the test to crude cottonseed oil, but he has experimented on a number of specimens of commercial oil, and refined oil and cottonseed stearine; his observation has been that the refined oil does not give as marked a color as the commercial oil does; he can not say whether or not there would be any difference in the oil made in summer or winter; he has concluded that the color comes from the oil itself, and not from any foreign substances that might be in it, because he has tested three specimens of fine oil, which were entirely colorless, and has also tested perfectly colorless cottonseed stearine, and from these tests he concludes the color produced by the test is due to the oil itself. In respect to the variation of the amount of stearine in different samples of lard, he accepts as evidence of its truth the agreement of authorities who have discussed it in books and other publications; he understands the agreement of writers, on this point, to rest on the well-known fact that in lard are the constituents of stearine and palmitine, which are solid oils; these can be separated approximately, not perfectly, perhaps, but sufficiently nearly so, when treated alike, to enable one to determine that some lards contain more stearine than others.

* * * * *

His attention was first directed to the subject of the detection of tallow in lard about five or six years ago; at that time he made some chemical experiments on the question, and again, about eighteen months ago, he made a few other experiments in the same direction, and within the past four weeks he has made numerous experiments. Husson's method, as published, is to take a mixture of alcohol, at 90 degrees, and ether at 66 degrees, which he understands to mean 90 per cent. alcohol and absolute ether; with this mixture he treats the previously warmed fat and allows the more solid portion of it to crystallize; he has used Husson's method, as he read it in French, and failed to obtain satisfactory results by it, and considered it untrustworthy; he does not fully condemn it, because he is not fully convinced as to what Husson means by alcohol at 90 degrees and ether at 60 degrees; if his interpretation of the method is correct, he does not concur in its being of value. The comparative results obtained by Professor Delafontaine and others, who have testified on behalf of the prosecution in this case, by which the lard in question was shown to have more stearine than the pure lard, which they used in comparison, carries no conclusion to his mind whatever, for the reason that Professor Delafontaine testified that the only lard which he knew to be pure, and with which he tested the samples in comparison, was kettle-rendered lard. It is manifestly unfair to take that as a standard for comparison with prime steam lard; but besides that, his own experience in treating lard, by the process described by Professor Delafontaine, shows it to be fallacious from the very foundation, and he attaches no importance to any results obtained by it or by modifications of it.

He means to be understood that, so far as he is able to determine, the samples of the lard now under consideration, which were examined by him, contain absolutely no adulteration.

CERTIFICATE OF PROF. E. B. STEWART.*

JULY 3, 1883.

This certifies that I have carefully examined three specimens of lard received from Prof. W. S. Haines on the 20th of June, 1883, and find that they present the characteristic of pure hog's lard, free from tallow and cottonseed oil.

E. B. STEWART.

METHOD OF EXAMINATION FOLLOWED BY PROFESSOR STEWART.†

Both pure kettle-rendered and steam-rendered lard were treated with about three times its weight of absolute alcohol at a temperature just sufficient to melt; the solid residuum which separated on cooling was assumed to consist of tristearate of glyceryl principally; this was treated with, first, oil of turpentine; second, petroleum naphtha; third, bisulphide of carbon; fourth, benzole; fifth, Squibb's strongest ether; and, sixth, melted in balsam fir. Pure beef tallow was treated with absolute alcohol in the same way, and subsequently with the same reagents.

E. B. STEWART.

JULY 3, 1883.

CERTIFICATE OF PROF. S. P. SHARPLESS.‡

JULY 3, 1883.

I have examined three samples of lard submitted to me by Prof. W. S. Haines, and marked Nos. 1, 2, 3, Fowler Bros. I have been unable to find any adulteration in these samples, and believe them to be pure hog product.

S. P. SHARPLESS.

In explanation of his results Professor Sharpless says :§

He received from Professor Haines, two weeks ago, three samples of lard, marked, respectively, No. 1, No. 2, and No. 3, since which date he has devoted his time to their examination. The samples were received in tin boxes, wrapped in paper, and properly sealed. The work of examination was commenced on the 19th of June, and was conducted jointly by Professor Doremus, Professor Haines, and himself, at the laboratory of the College of the City of New York. After opening the boxes the contents of each were thoroughly mixed, and then 5 grams were weighed out from each of the boxes; these specimens were submitted to the action of absolute alcohol and the strongest ether, both being carefully tested as to their strength; he, at the same time, for the purpose of comparison, prepared a sample of tallow, rendering it himself in order to be certain of its purity; 5 grams of this tallow were also weighed out; these samples were weighed into small flasks, and the fats of each were melted, and then 50cc of the mixture of alcohol and ether was poured over each specimen; the flasks were shaken until the alcohol and ether had completely dissolved the whole of the fat. This process is a test that will show whether the lard contained starch or salt or whether there is much water in it; lard having much water in it will give a clear solution, but will be milky in appearance; these lards all gave perfectly clear solutions, with perhaps an occasional particle of wood from the cask; there was very little fibrous matter in any of the samples; he has never yet seen samples of lard that were perfectly free from fiber; these were as free from it as any he had ever seen; the flasks were set in a closet, at the same temperature for each, until the next morning, when they were examined. The bulk of the precipitate in the different flasks differed; in some it was slightly flocculent, in others it adhered to the bottom of the flask; this latter condition was more marked in the case of this tallow, which formed a thin layer over the bottom of the flask; the liquid was poured off and 25cc of fresh was added, and the flasks were allowed to stand, with the fresh solvent in them, until the next morning, and

* Op. cit., p. 228.

† Op. cit., p. 228.

‡ Op. cit., p. 233.

§ Op. cit., pp. 229-230.

then their contents were filtered, the precipitate washed with a little absolute alcohol, and weighed. Sample No. 1 gave 4.35 per cent. of precipitate; No. 2 gave 2.99; No. 3 gave 2.4, and the tallow gave 5.3 per cent. of precipitate. At the same time another series of experiments was tried by weighing out the same amount of lard and adding to it the mixture of alcohol and ether; in this case the lard was not melted previous to the addition of the alcohol and ether; after these were added the flasks were shaken thoroughly and stood in a water-bath at the temperature of Croton water, which at that time was about 72° F., the water being allowed to run around the flasks; they were left standing in the water in this way for a little over two hours, during which time they were shaken every fifteen minutes; at the end of this time the contents were allowed to settle; the liquid was then poured off and 25cc more of the solvent was added, again shaken, and the residue of each sample was collected on filters, which had previously been weighed; this series of tests produced a little higher per cent. of residue in every case than the former. No. 1 gave 5.63; No. 2 gave 4.17; No. 3 gave 3.69, and it was found impossible to filter the pure tallow on account of the large mass of crystals it gave. The day after this test they received a sample of pure prime steam lard from Chicago, rendered under the supervision of Dr. Tilley; he made two parallel experiments upon this pure lard; the first one gave a residuum of .44 of 1 per cent.; the second gave 3.14 per cent. These samples were taken from the same mass of lard at the same time, and were treated with exactly the same solvent of alcohol and ether; during the process they stood side by side in the same closet, were filtered off at the same time, weighed at the same time, and in every way treated alike, and yet one gave nearly eight times as much residue as the other. In connection with Professors Remsen and Witthaus he tried another series of experiments, following out the same method as in the first series, with results substantially the same as in the first experiment, which he has detailed so far as the lard was concerned. In this experiment there was also a mixture of lard and tallow, but he did not find that the addition of the tallow made any perceptible difference in the result. This experiment will probably be more fully described by Professor Remsen.

CERTIFICATE OF PROF. IRA REMSEN.*

CHICAGO, July 5, 1883.

I hereby certify that I have examined by chemical methods the three samples of lard designated as Nos. 1, 2, and 3, "Fowler," submitted to me by Prof. W. S. Haines, and have failed to find any foreign substance in them. I am, therefore, of the opinion that the samples are pure lard.

IRA REMSEN.

Professor Remsen† stated that he received from Prof. W. S. Haines, for analysis, three samples of lard, designated 1, 2, and 3, "Fowler," and has been working on them constantly for about ten days, to a considerable extent night and day. When these samples were first submitted to him he set about a very careful search through the literature on the subject to determine what method ought to be adopted in the examinations of them. He was disappointed by finding that the chemical study of lard had, apparently, received very little attention. The methods for the chemical examination of lard which have been, perhaps, the most frequently employed, are similar to those which are used in the examination of butter; indeed, the chemical knowledge of butter is much more general than that of lard. After considering the subject he decided, as he thinks most do who are called upon to investigate lard, to adopt as an experimental method that of Husson, which is based on a very simple principle. Fats are known to differ in the proportions of oleine, palmitine, and stearine contained in them. In liquid fats there is a larger proportion of oleine, and a less proportion of stearine than in those of a more solid character. In case a fat

* Op. cit., p. 246.

† Op. cit., pp. 238, 239, 240, 241, 245, 246.

which is naturally rich in stearine should be adulterated with one which is rich in oleine, he thinks chemistry could very easily detect that adulteration; there are, however, several fats which are so similar to each other, in respect to the proportions of the constituents they contain, that when they are mixed it is a very difficult matter for chemistry to detect the mixture. The best method he could think of was the separation of the stearine from the oleine, if that could be effected; but there is no method by which this can be entirely or anything like entirely done. If fats are treated, in comparison, with some substance which will partly separate the stearine, but leave some behind, and then that which is left behind in the one is compared with that left behind in the other, both being accurately ascertained, and one is found to be much larger than it should be, it is strong ground for suspicion in one's mind that there is something the matter with it, without, perhaps, being able to say exactly what has been put into it. In order to test a sample of lard by this process it must first be known what standard lard really contains, or, if tallow or any other fat, what that fat contains; this, however, can not be ascertained from the books, because, in respect to reliable data on the subject, they are singularly silent. It is stated repeatedly, in the books, that lard varies in its composition, depending on a variety of causes, such as the dryness and other conditions of the food on which the animal from which it was produced was fed, the season of the year when fed or when killed, etc. No one seems to have made so complete an investigation of the subject as to state to what extent these variations may go, hence it is absolutely necessary, in an examination of lard, to first get something that may be considered a standard of pure lard, to know what is pure lard, and to make an exhaustive investigation of the subject. There ought to be a very extensive investigation of different specimens of lard, so as to find out what variations in the constituents are possible.

For the purposes of this investigation, he procured at the outset a sample of lard known to be pure, with which comparisons could be made as the investigations progressed; and then if, on a comparative examination, the lards submitted for examination were found to conduct themselves in all respects in the same manner, and no differences were found in them, the conclusion would be justified that the lards to be tested were pure. In the process of examination, he in the first place applied Husson's method, and also a modification of that method, suggested from reading the testimony of Professor Delafontaine. He also applied the elaidine test and the pattern test, and he treated them with sulphuric acid. He also examined these samples by means of the spectroscope and by transmitted light, and he has to some extent examined them microscopically; and he can say that, after all the examinations which he has been able to give to the samples of lard submitted to him by Professor Haines, his only conclusion is that he can find no impurities whatever in them.

In his examinations he has paid particular attention to the method described by Professor Delafontaine, in that gentleman's testimony in this case. That is not the method described in the books, and known as Husson's method, but is a modification of that method to suit the case of lard. It depends upon the relative amount of residue remaining after treatment with alcohol and ether, and he has to say that if that method is a good and reliable method, then, beyond any possible question in his mind, the samples 1, 2, and 3 submitted to him are pure lard. If it is not a reliable method it proves nothing. He is not prepared to absolutely condemn the method, for the subject has never been studied, so far as he knows, with that care that would warrant the basing of positive conclusions upon that process of determining it. It is possible that there is the germ of a good method in it; but, as described by Professor Delafontaine, he is quite confident it could never be used to prove positively whether lard is pure or impure. In respect to Husson's method, without any modification, he should say most emphatically that it is not a reliable process for determining the adulteration of lard. The Blythe pattern process is, as he understands it, the same as what is known as "cohesion figures." There is some confusion in the use of terms in describing matters of this sort. He is, however, quite sure that the process called the

"Blythe pattern process" and "cohesion figures" is one and the same thing. . Of this process Mr. William L. Carpenter, in the "Journal of the Society of Chemical Industry," of London, under date of March 29, 1883, says:

"In reply to Mr. Newland's inquiry on this subject, I may say that when Professor Tomlinson first brought them forward I spent several weeks in fruitless endeavors to apply the method to analytical examination of oil, and the result, I regret to say, was a complete failure."

He has read the testimony of Mr. Hoskins given in this case in respect to his chemical analysis of lard. Mr. Hoskins washes out a little differently from Professor Delafontaine, but he regards Mr. Hoskin's process very much as he does that of Professor Delafontaine. He has also read the testimony of Mr. Hirsh, and will say that he considers the chemical process pursued by Mr. Hirsh as the least reliable of any of those referred to. Mr. Hirsh made use of the same principle that the other two chemists named did, or attempted to do so. Mr. Hirsh, taking the same quantity of the lard to be examined and of pure lard, and applying his process, seeks to compare the residue obtained in each specimen; that principle is the basis of the methods of all these gentlemen; the others weigh the residue obtained, which is the only possible way to deal with it chemically, but Mr. Hirsh measures it; he has never before heard of measuring a precipitate; that is something entirely novel and original.

In prosecuting this investigation he tried the color test for cottonseed oil, with sulphuric acid; this consists of taking a known quantity of the specimen to be examined and dropping on it two drops of sulphuric acid; with cotton-seed oil the effect of this combination is to produce a change of color. This method was tried with cottonseed oil, with mixtures of cottonseed oil and lard, one of which was as low as 10 per cent. of the oil, and with the samples 1, 2, and 3 (Fowler lard); in the mixture of lard with 10 per cent. cotton-seed oil, he could positively identify the presence of the cottonseed oil, but he could not detect any evidence of it in the samples 1, 2, and 3. This test depends for its success upon having the right conditions; it is an extremely delicate test, and must be made under certain conditions in order to get any results at all. He has since tried the same process with another sulphuric acid and failed in being able to distinguish one from the other; that experiment did not prove anything to his mind.

The subject of investigations for the detection of cottonseed oil or tallow in lard is one of the most complicated with which chemists have to deal. When chemists say they can not solve such questions people are apt to laugh at them. Altogether too much is expected of chemistry in some cases, and in others not enough of credit is given. The subject of investigation of fats has been worked on for many years, and all the methods which have been employed have been, in general, found unsatisfactory. In the case of butter the question has, within the past ten years, been studied with great care, and in consequence it is now possible to tell positively what the nature of butter is; other fats have not been examined with the same care, owing to the immense amount of time and labor necessary to go through the investigation fully. In a general way he will say that the methods employed for determining the question are unsatisfactory to him. In the case of the samples 1, 2, and 3, now in question, he can say, that with the investigations he has been able to make of them he has found no evidence whatever of impurity. All the tests he has applied to them, so far as they have given indications, have indicated the absence of impurity; the methods being imperfect, he can not say positively that the lard is pure, but the indications are all in that direction, with no indications whatever in the opposite direction.

* * * * *

The spectroscope develops a difference in the appearance between cottonseed oil, either alone or mixed with lard, and that of pure lard; by the use of the spectroscope he was able to positively, and without difficulty, tell that a certain specimen, unknown to him at the time, but which really contained 10 per cent. of cottonseed

oil, had cottonseed oil in it. The samples of Fowler lard did not give the cottonseed oil appearance when tested by the spectroscope, but acted in all respects the same as the pure prime steam lard did. He does not attach much importance to this test, and can only say that so far as the examination of the Fowler lard by this test is concerned, the results were negative.

When examined by transmitted light cottonseed oil has a yellowish color, which neither pure lard nor tallow has, but all mixtures of lard and cottonseed oil have this color. The Fowler lards failed to show any appearance of this color when examined by transmitted light.

The cottonseed oil he used in all these experiments was refined oil. He has never used the bleached or colorless oil for such experiments. The test by transmitted light would be of no value whatever in detecting the colorless oil.

Finally, in respect to the color test for the detection of cottonseed oil: When he testified two days since, he had spoken cautiously and not very confidently of the value of this process. At that time he had just come from the laboratory where they (himself and others) had met with a difficulty which at that time none of them could solve. This arose from their having used something they did not know anything about. This difficulty has, however, been since explained, and he is now fully prepared to make a positive statement in regard to the value of the sulphuric-acid test as a means of detecting an admixture of cottonseed oil in lard, and has joined with Professor Sharpless and others in the statement read by Professor Sharpless in that gentleman's final testimony in regard to the value of that test. The expression of this paper (see pp. 233, 234, which the witness read) is his deliberate judgment upon the question of the reliability of the color test for cottonseed oil; and in view of all his examinations of the samples 1, 2, and 3 of Fowler lard, he is now prepared to express a positive opinion that these samples do not contain any cottonseed oil. He did not, as he has said, use any bleached oil in this process, but other gentlemen did, who have given, or will give, evidence on that point. In all the oil he has used he always gets the color reaction peculiar to cottonseed oil, not always to exactly the same extent, but so sufficiently and clearly marked as to be unmistakable, and the absence of it is proof positive, to his mind, that there is no cottonseed oil in the samples of Fowler lard he has examined.

CERTIFICATE OF PROF. R. A. WITTHAUS.*

CHICAGO, *July 5, 1883.*

This is to certify that I have made chemical examinations of three samples of lard marked, respectively, Nos. 1, 2, and 3, Fowler, without obtaining the slightest evidence of the presence of any impurity. I therefore consider them as being samples of pure prime steam lard.

R. A. WITTHAUS.

Professor Witthaus also exhibited a table showing the results of five experiments with modifications of Husson, showing the varying proportions of insoluble residue left by ether and alcohol. In the first four experiments the residue was washed with 10cc of absolute alcohol, in the fifth with 30cc.†

* Op. cit., p. 254.

† Op. cit., table, p. 249.

Substances treated.	Per cent. of residue.				
	No. 1.	No. 2.	No. 3.	No. 4.	No. 5.
Fowler's lard :					
No. 1	5.44	5.06	4.65	3.94
No. 2	4.03	3.38	3.18	2.88
No. 3	3.64	3.12	2.68	1.80
Prime steam lard :					
From the can	3.64	3.27	3.85	3.19	1.69
From the tierce	4.02	3.35
Pure tallow	4.22	3.24	3.79	2.46
90 per cent. pure lard, 10 per cent. tallow	3.42
80 per cent. pure lard, 20 per cent. tallow	3.54
70 per cent. pure lard, 30 per cent. tallow	3.58
60 per cent. pure lard, 40 per cent. tallow	3.28
90 per cent. pure lard, 10 per cent. cottonseed oil	2.67
80 per cent. pure lard { 10 per cent. cottonseed oil .. } { 10 per cent. tallow	2.56
60 per cent. pure lard { 10 per cent. cottonseed oil .. } { 30 per cent. tallow	2.23

The last experiment (No. 5) was tried to ascertain the effect of washing the residue with 30 instead of 10cc of absolute alcohol. The result clearly showed that much depends on the amount of washing to which the residue may be subjected. This residue is not absolutely insoluble in absolute alcohol, and it is probable that by excessive washings it might all disappear.

It does not appear from Professor Delafontaine's testimony that he washed the residue at all; but, inasmuch as some of the liquid containing more or less of the dissolved material, would remain on the filter, unless washed off by the alcohol, and drying, would improperly increase the weight of the precipitate, they deemed the washing with a very small quantity of alcohol necessary in order to arrive at true results. He should not expect to get essentially different comparative results by not washing, and as the whole experiment is comparative it makes little difference whether the washing is done or not.

Professor Delafontaine claims that the presence of tallow is proved by an increase of the residue. The results of the experiments shown in the table prove exactly the contrary, so far as they prove anything in that respect. The admixture of cottonseed oil tends to greatly reduce the amount of the residue obtained by Professor Delafontaine's method.

CERTIFICATE OF W. M. HABERSHAW.*

CHICAGO, July 5, 1883.

I have analyzed three samples (sealed) of lard marked Nos. 1, 2, and 3, delivered to me by Dr. W. S. Haines on the 21st of June, 1883, and find them free from adulteration, and, in my opinion, pure lard.

HABERSHAW.

Mr. Habershaw said: †

The analysis of fats has, until the last five years, been a question involving a great deal of doubt. Among the first published methods of treating fats was the color test,

* Op. cit., p. 258.

† Op. cit., pp. 257, 258.

described in a French work by Theodore Chateau. By that test oils and fats were treated with different reagents for producing different colors. He has examined all kinds of oils by that method. In some cases good results are obtained, in others they are unsatisfactory. The method is of somewhat doubtful value. Then came the elaidine test, by which the oleine of a fat is hardened by the action of an oxidizing agent. He does not regard that test as of any value. About 1878, a German method for analyzing oils by means of a standard solution of an alkali was published. He has used that method from then until now, and has found it to produce excellent results. In that method the substance is accurately weighed and treated with hydrate of potash of known value. The result is expressed in milligrams or grams of hydrate of potash required to saponify a stated amount of fat. The standard which is used is 1 gram of fat, equivalent to (blank) milligrams of hydrate of potash. In conjunction with that process, if an oil be examined by the oleate of lead process, which enables one to separate the equivalent oleate of lead from the equivalent stearic and palmitic acid, you ascertain the amount of the stearic and palmitic acids derived by difference, or they may be estimated directly.

* * * * *

His work has been entirely independent of other chemists who have examined those samples of lard. The composition of lard is about 47 of oleic acid and 47 of stearic acid: he can not give the chemical formula of lard; the lowest amount of stearine he has found in pure commercial lard was 38 to 40 per cent.; the highest amount was about 45 per cent.; by stearine he means the combination of stearine and palmitine; he has never analyzed lard so as to obtain the tristearine, and has never made the ultimate analysis of either oleine, palmitine or stearine. He does not think there is any difference between the olein of lard and that of tallow; the chemical characteristics of chemically pure stearine are always identical; in the mixture of stearine and palmitine known to the trade as stearine the characteristics would, he supposes, differ, but he can not describe the differences. He believes in the sulphuric-acid test for the detection of cottonseed oil, when used by those who understand it; he has had a great deal of experience with that test and he can detect cottonseed oil by it. He has made a great many analyses of butter; and he has used the Angell and H  hner process, with which he is quite familiar. In the analysis of butter the question of its purity is decided by its percentage of insoluble fatty acids; his own analyses give 87 per cent. as the average of fatty acids in butter; Angell gives 87.34; the range is 2 per cent. either way; if a sample of butter runs over 1 or 2 per cent. above the maximum he has found in pure butter he would condemn it.

TESTIMONY OF PROF. R. OGDEN DOREMUS.*

Professor Doremus said he undertook the analysis of the samples 1, 2, and 3, Fowler, and the samples of pure lard by what is called Mutter's process, which consists in precipitating the oleic, palmitic, and stearic acids by a salt of lead; this gives the oleate of lead, the palmitate of lead and the stearate of lead; the oleate of lead alone is soluble in ether. After the oleate of lead was removed by this solvent and after filtration, it was decomposed by an acid, and a solution of oleic acid and ether was obtained. A small part of this solution was drawn off, the ether evaporated, and the residue weighed; from this the amount of oleic acid was estimated; the palmitate of lead and the stearate of lead which remained in the filter were removed, decomposed by an acid, and weighed, giving the palmitic and stearic acids combined. Chemistry has not reached that degree of perfection by which these two last-named acids can

* Op. cit., pp. 261, 262, 263, 254, 265.

be completely separated; therefore they must be estimated in combination; the results of this process were as follows, in percentages:

Acids.	No. 1.	No. 2.	No. 3.	Pure lard.
Oleic acid.....	60.60	60.42	65.02	65.40
Palmitic and stearic acids	33.85	31.88	30.42	28.16
Total	94.45	92.30	95.44	93.56

The remainder of the substance was glycerine, which being soluble in water was washed away. Lards are liable to variation in the proportion of these acids which they contain, owing to various causes, such as differences in feed of the animals, different seasons of the year in which they are killed, and other causes, and there are also differences in the lard taken from different parts of the same animal. From the fact that the analyses of all these specimens so nearly agree in their proportions of the acids—the variations being only such as are liable to be found in pure lard—he feels justified in stating that he believes the samples, Fowler Nos. 1, 2, and 3, contain the proper proportions of the ingredients of lard and are, therefore, pure.

Analytical chemistry is not capable of determining whether a specimen of stearine is from the fat of the hog or from the fat of the bullock, but these lards, being shown to contain the proper proportions of the constituents of pure prime steam lard, he claims are pure. Muter, as he now remembers it, reports lard as containing a little over 47 per cent. of oleine and about the same percentage of palmitine and stearine, but Muter doubtless referred to lard rendered from the leaf fat alone; in Europe they would not, as we do in this country, designate as lard the fat from all parts of the hog but only such as comes from the leaf.

He has examined under the microscope the specimens of crystals obtained by Dr. Belfield, Professor Hayes, and others, and he believes the microscope is capable of determining the question as to whether the substance from which the crystals were obtained was the stearine of beef or of the hog. He does not claim to be a professional microscopist, but he has used the microscope largely in chemical investigations and in instruction to college students. In examining the crystals of Professor Hayes, slide after slide was placed before him, under the microscope, and without any previous knowledge on his part as to what the specimen was, he was able to at once correctly decide which was from pure lard, which from tallow, and which from mixtures of the two. The difference in the crystals is very marked, and is beautifully illustrated by the photographs exhibited by Dr. Belfield.

In respect to adulteration of lard by cottonseed oil, he believes that gentlemen who are skilled in handling these substances can by the ordinary senses of taste and smell, and by its color, detect an adulteration by it when the adulteration amounts to 5 or 10 per cent., and if the adulteration has been with the common or unbleached oil, he may not, from the color, be able to say that it is certainly cottonseed oil that has been mixed, but he will be able to detect the presence of some abnormal substance. He believes that an expert can, by the sulphuric-acid test, decide whether lard is adulterated with cottonseed oil. There are two methods of doing this. First, dropping the acid upon the lard and allowing it to remain, watching the changes of color. Second, dropping the acid upon the lard and stirring them together, and then watching the development of colors. By this process an adulteration by a substance like cottonseed oil can certainly be detected if it exist to the extent of 10 per cent. He has read the testimony for the defense in this case on the subject of the sulphuric-acid test, and fully agrees with them as to the value of this test, and he has joined in the statement in that regard read by Professor Sharpless.

By the aid of the spectroscope for the examination of lard, cottonseed oil, if present, is at once and clearly indicated; not that it is certainly cottonseed oil, but that

there is some foreign substance in the lard. There is nothing in chemical analysis that can compare in delicacy with the spectroscope; the utmost reliance is placed on this instrument, and it is used for the determination of the most serious and delicate questions, and such as involve the issues of life and death. There are two forms of this spectroscope, that of direct vision, and the micro-spectroscope; the latter being a combination of the microscope and the spectroscope; both were employed in the examination of the samples of the lard now in question. A piece of rubber was arranged with small cavities cut into it, and into one of these cavities was placed a specimen of pure lard, which had been previously melted and filtered; in another cottonseed oil; in another lard with an admixture of 10 per cent of cottonseed oil; in another lard mixed with 20 per cent. of cottonseed oil (the lard used in all these specimens had been filtered) and also one specimen of lard very carefully filtered; the cottonseed oil had not been filtered. These several samples were placed in front of the spectroscope so that the light would pass through the liquids in succession. The observation showed that in the case of the pure filtered lard there was a very trivial obscuration of the whole spectrum; in the more carefully filtered lard scarcely any, as the light passed through them; in the sample of pure cottonseed oil the whole of the upper part of the spectrum, from the upper or blue end down to the space between Fraunhofer's lines E. and F., was obliterated. A second prism was then adjusted so that the light from another source could pass through the spectroscope, revealing one spectrum above another. These lights were so adjusted as that both had the same degree of brilliancy. In this way they could observe in one spectrum the light passing through unobstructed and in the other passing through the specimen. Examined in this way the filtered pure lard appeared as clear and brilliant in the one spectrum as the unobstructed light did in the other; no difference could be detected. The pure cottonseed oil being brought in the place of the pure lard, only one-half of the spectrum could be seen, the part only through which the light passed unobstructed, the other half being entirely obliterated. The 10 per cent. mixture of cottonseed oil produced a very perceptible obscuration; the 20 per cent. mixture much more, so that an approximate estimate can be made by the degree of obscuration; it would, perhaps, be a rough estimate, but you can certainly say whether it is present or not. The samples of Fowler lard 1, 2, and 3 were, after being heated and filtered, subjected to this test, and examined in the same way; with them there was not the slightest obscuration of the blue end; the one spectrum had the same brilliancy as the other. He has not examined a great variety of oils with the spectroscope, and can not say what, if any, other substance would similarly affect the light. Olive oil affects it differently; that produces a dark band on the lower part of the spectrum. These experiments were specially with reference to the detecting of cottonseed oil, and his experiments have been sufficient to warrant him in claiming that he can certainly detect 10 per cent. of cottonseed oil mixed with lard; he tried it with an admixture of 5 per cent., and found some obscuration; his son claimed he could detect 5 per cent. every time, but he (the witness) will not assert that he can do that, but he can a 10 per cent. mixture; he does not claim to be able to state that the adulteration is certainly cottonseed oil, but where there is no obscuration he will say that cottonseed oil is not present, at least not to the extent of 10 per cent.; with practice, he thinks it probable that a much less adulteration, by cottonseed oil, than 10 per cent. can be detected with certainty. It is necessary that the lard should be melted and filtered, so that all the particles of membrane can be removed from it. If melted and not filtered, there will be a slight obscuration, but it will affect all parts of the spectrum alike; if the lard is properly filtered there will be no obscuration whatever; the presence of cottonseed oil affects the blue end only, and the degree of obscuration, from a slight darkening to a total obliteration, depends on the amount of the cottonseed oil present in the specimen. The micro-spectroscope developed the same effects as the spectroscope alone, in respect to cottonseed oil.

He has tried the elaidine test, but has not been able to successfully use it in detect-

ing cottonseed oil. He has also, repeatedly, tried the Blythe pattern process, but has not, by it, been able to reach any results upon which he could rely.

The testimony for the defense being all in, Dr. Delafontaine was called in rebuttal and made the following statements: *

Prof. M. Delafontaine recalled by the prosecution in rebuttal of statements and theories presented in the evidence of witnesses for the defense, testified that it was evident to him that the scientific witnesses for the defense could not have read his evidence with care, or they would not have charged him with using kettle-rendered lard alone as his standard sample for comparison, as he had distinctly stated that he got a sample of prime steam lard from a packing-house, which, on being tested in the same way as the others, gave about 3 per cent. of residue, and he had added, that, as a clincher, he had taken lard stearine, 1 pound of which is equal to 2 pounds of lard, and found that it did not run higher in residue than his sample, No. 1, of Fowler lard. In other analyses of four samples of prime steam lard of undoubted purity, none ran higher than 3 per cent. of residue. The chemists whose evidence on behalf of the defense, while seeking to impress the board with the unreliability of the process he described in his evidence in chief, all admit they did not, in their attempted trials of that process, follow the process he described, neither faithfully nor closely, as they should have done; all introduced some modifications, some of which are essential, and entirely change the character of the method; others may be of small importance; he can not say whether they are or not. There seems to be a general disposition to raise all sorts of objections to his process; apparently, in the hope that some of them would stick. The gentlemen from the East acknowledge that they have very little knowledge of chemistry of fats; for instance, Professor Doremus says the composition of fats varies a great deal; and reported that he had found 65 per cent. of oleic acid (which means 68 per cent. of oleine) in pure lard; and he (Professor Doremus) says Muter found 47 per cent. of oleine in lard, and claims there is that range of variation in pure lard. Professor Doremus has never analyzed but one sample of pure lard, while he (the witness) has analyzed many, and knows better than Professor Doremus does within what limits pure lard varies. The sample which Muter analyzed was, in all probability, refined lard; he himself, some two years or so ago, analyzed some Chicago refined lard, and found only 48 per cent. oleine; it was mixed with tallow and other things, such as are put into refined lard.

He regards Professor Doremus's reported analysis of four samples of lard—two of which yielded about 95 per cent. of fatty acids, and the other two about 92, or 92.5 per cent. as faulty; because it has been recognized since 1816 that the yield of fatty acids in various fats is nearly the same in all—between 95 and 96 per cent. The man who has done most to enlighten us in the knowledge of fats finds that, "in human fat the total amount of fatty substance in 100 is 95; in mutton fat, 95.5; in beef fat 95; in pork fat 94.9." He (the witness) claims that whenever a chemist finds even 1 per cent. less than these figures he should conclude his analysis is not correct.

He thinks that the explanation of the fact, if it is a fact, that by his process these gentlemen got a less yield of residue, from mixtures of lard and tallow, than from pure tallow alone, is in the time the substances remained mixed before being analyzed; that might make an essential difference. In his tests he took samples of the Fowler lard, and of pure lard, and added to each a quantity of beef stearine; the result, in both cases, was that a residue was obtained equal to what was in the lard, plus what was in the beef stearine. In the case of the elaidine test, all of the gentlemen say it gives no results, but none of them applied it as he testified he did; all had some modification—some used nitric acid.

He has tried the sulphuric acid color tests on the Fowler samples of lard, and got those colors the gentlemen spoke so enthusiastically about, but in treating by that

test he separated the oil, and tested it, which they did not do; if they had done this they would have got the cottonseed oil colors they say was absent in their treatment of it. The cottonseed oil becomes so diluted when mixed with the mass of lard that it is more difficult to detect it in the lard itself than in the separated oil. He did not get the color from the Fowler lard, but he did get it when he treated the oil from that lard alone. He has tried this test recently on the oil from a sample of lard which contained cottonseed oil, and he got the colors they describe and then tried it on some prime lard oil and got no such color. The color test by sulphuric acid, if the oil is treated, is a valuable test for cottonseed oil, but his experiments show that the cottonseed oil may be so well refined that it will not answer to the test quite so well as for oil in the less refined state.


He has tried the process of obtaining crystals for microscopic examination by a solution. He took a sample of the Fowler lard and a sample of pure lard for a comparison, and treated them by that method, and after four or five hours, when half the ether was evaporated, there was in the Fowler lard an abundant deposit of crystals, in the pure lard none yet; he examined those crystals from the Fowler lard, and found them to be nearly all crystals of stearine, with a small sprinkling of crystals of palmitine; the sample of pure lard was allowed to stand four hours longer, the ether then being reduced to one-quarter of its original bulk; from this he got a small crop of crystals; on examining these by the microscope he found them to be mainly crystals of palmitine, with some of stearine; this process leads to determining whether the crystals are those of stearine or of palmitine, when it is carefully applied; the gentlemen on the other side all acknowledge they do not know the difference between the crystals of stearine and those of palmitine; his treatment by this process showed that the Fowler sample of lard had a great deal more stearine in it than the pure lard had.

He thinks the gentlemen have greatly exaggerated objections to the method pursued by Mr. Hoskins in testing by the pattern process; he has seen that process tried on samples of lard and of tallow, and on mixtures of lard and tallow, and found quite different patterns produced in these specimens, when the process was properly applied.

He has tried Husson's method, but did not get satisfactory results from it; he had no difficulty in understanding what was meant by 90 degrees alcohol and 66 degrees ether; both mean the degrees measured by the hydrometer, and are the equivalent of per cent.; it is a quite usual mode of expressing the strength of such substances in French.

DECISION OF THE BOARD OF TRADE.*

The board find that the charges preferred may be properly summarized under the following general heads, to wit:

First.—That a certain lot of 250 tierces of lard, manufactured by the Anglo-American Packing and Provision Company, branded “James Wright & Co. Prime Steam Lard” and marked “ 10,” which lot of lard was stored in a provision warehouse of the Anglo-American Packing and Provision Company, and represented by a warehouse receipt issued by said company—having been by it put upon the market and sold as and for prime steam lard—was delivered to complainants in the course of business and paid for by them as prime steam lard, but was not in fact prime steam lard, as required by the rules of the Board of Trade, but was adulterated and contained substances other than hog lard, to wit, tallow, vegetable oils, etc., as was said to have been stated by competent and skilled chemists who had analyzed the same, and as said complainants charge and believe to be the fact.

Second.—That on the first day of June, 1883, said Fowler Brothers tendered to said complainants a certain lot or lots of lard—brands and marks, other than prime steam lard, not stated—which tender purported to be in fulfillment of a contract made by

* Op. cit., pp. 270-272.

said Fowler Brothers to deliver to complainants a large quantity of prime steam lard, then deliverable on said contracts, which said lard, complainants charge, was not in fact prime steam lard, as required by the rules of the Board of Trade, but was mixed and adulterated by and for said Fowler Brothers, with tallow, beef fat, cotton-seed oil, or other substance different from hog's lard; which tender was intended by the said Fowler Brothers to deceive, defraud, and cheat complainants by delivering to them a spurious commodity under the brand and name of prime steam lard.

Third.—Complainants charge upon information and belief that the Anglo-American Packing and Provision Company, with the knowledge and consent of said Fowlers, has manufactured a large quantity of adulterated lard and mixtures which has been sold by said Fowlers to the trade, and to members of the Board of Trade, for prime steam lard, which said adulterated lard is stored in the warehouses under their control, and which transactions complainants charge to be acts of bad faith and dishonorable and dishonest conduct in business.

The board of directors have given to the investigation of these charges a very protracted and patient hearing, which in their judgment has been exhausted in developing all the facts attainable in respect to them, and have arrived at the conclusion that they have not been sustained, and have therefore voted that they be dismissed.

Inasmuch, however, as these charges involve questions of the greatest concern to the members of this association, and to dealers and consumers of pork products, not only throughout our own country but in foreign lands as well, the board of directors, in view of the evidence submitted in this case, both on the part of defendants and for the prosecution, can not, with a due regard to their responsibilities to the public and to the members of this association, refrain from expressing their unqualified disapproval of and censure upon defendants for the remarkable methods of conducting the business of manufacturing lard in their establishment, as developed by the evidence in this case. It appears, and is admitted, to have been the practice, during at least several of recent months, that beef product in various forms has been rendered in the same tanks and with hog product, this mixed product of certain tanks being conducted through a system of intricate machinery and pipes in which also prime steam lard was at times conveyed to their so-called lard refinery wherein both prime steam lard and the mixed product used for what is called refined lard is drawn off into packages for market; and this in a manner that by accident or design on the part of the employes of the establishment could easily contaminate the purity of their prime steam lard, which might thus become more or less adulterated, not only with the beef product so rendered with a portion of their hog product, but also with the cotton-seed oil and other unknown substances used in the manufacture of their so-called refined lard; and this board, in view of the existing methods of manufacturing prime steam lard in this establishment, recommend that, without delay, the parties so readjust their lard-manufacturing arrangements that all grounds for suspicion in this respect shall be effectually removed, and that in case this recommendation is not promptly complied with to the satisfaction of this board, such action be taken as will relieve this board of all responsibility in respect to such product.

The board of directors would embrace this occasion to express their gratification that, as the result of this investigation, the question of ascertaining the truth as to adulterations in lard by scientific examination, which has hitherto, to say the least, been one of extreme difficulty, seems now to give promise of a satisfactory solution; and while not desiring to express absolute confidence in any particular method for determining adulterations by the substances suggested in the charges preferred in this case, the board feels great encouragement to believe that even small adulterations with cotton-seed oil can be detected by some of the methods detailed in the evidence submitted in the case by scientific gentlemen; and that the microscope, in the hands of an experienced operator, can be successfully employed in detecting adulteration by beef product when it exists to the extent of 10, and probably even a much less percentage.

ADULTERATION OF AMERICAN LARD.

IMPORTANT PROSECUTIONS.*

At the Liverpool police court on the 20th ultimo, before Mr. Raffles, several wholesale provision merchants in Liverpool were summoned for having sold lard not of the nature, substance, and quality demanded by the purchaser. Mr. Marks appeared to support the summonses on behalf of the Health Committee of the Corporation. The first case called was one in which Cuffey Brothers, Victoria street, were summoned, and for whom Mr. Pickford appeared. The court was crowded with representatives of the provision trade.

Mr. Marks, in opening the case, said the defendants, who carried on business at 40 Victoria street, were summoned for selling lard which was not of the nature, substance, and quality demanded by the purchaser. The warehouse of the defendants was visited on the 14th May by Inspector Baker, an officer under the sale of food and drugs act. He there saw a number of buckets of lard on which was printed "N. K. Fairbank & Co., refined lard, Chicago." He inquired the price, and ultimately purchased a bucket for 10s. 8d. He gave the usual notice about requiring the purchase for analysis, and offered to divide it for that purpose. His offer was accepted. He then left the defendants a sample, another portion he took to Dr. Campbell Brown, in the usual course, and the third he retained. Dr. Brown furnished a certificate, upon which, as a rule, the case rested. Owing, however, to communications that had been made to him (Mr. Marks) by a gentleman instructed on behalf of the defendants, in this and other cases, it was thought desirable that Dr. Brown should be in attendance, so that he might give evidence in a more ample manner than would appear from his certificate, and in order that the defendants might have an opportunity of cross-examining him. The certificate which Dr. Brown had furnished stated that he had analyzed the lard, and that in his opinion it contained considerably more than 40 per cent. of a mixture of cotton seed oil, and either mutton or beef fat. The court would probably gather this was a case of greater importance than cases under the sale of food and drugs act usually were. Certainly if a court was to be troubled with all the information which had been furnished to him officially, and also anonymously, he should imagine that, so far as the United States was concerned, the people were at present given up entirely to the lard question. [Laughter.] However, in the provision trade the case was undoubtedly regarded as of very great importance, and he was bound to say it was important to more classes of persons than one. It was of vast importance to the makers of lard, whose profits were simply enormous, and also to the consumers of the lard. It seemed that about eighteen months ago it was discovered that lard was being imported into this country which was adulterated. It was imported from America, and the fact appeared to have become known in America, and to have created a tremendous amount of feeling there.

Mr. Pickford objected to these observations as being irrelevant.

Mr. Marks said he was simply leading up to the facts. What he was going to say was, that the principal manufacturers of this lard were persons whose names appeared upon the buckets, namely, Fairbank & Co., of Chicago, and Armour & Co. It was known to Dr. Campbell Brown, about eighteen months ago, that this importation of lard was going on.

Mr. Pickford again objected to Mr. Marks entering into matters not connected with the present case.

Mr. Marks contended that his observations had reference to the case before the court.

Mr. Raffles said he should rule that what Dr. Brown did eighteen months ago was not relevant, except it had reference to the summons now being heard.

*The Analyst, July, 1888, pp. 136 *et seq.*

Mr. Marks (continuing) said that in order to meet Mr. Pickford's objections, he would put the matter in this way: On examining the sample in question, Dr. Brown found that his researches, which had occupied him more than eighteen months ago had furnished him with knowledge that had enabled him to discover that the sample contained cottonseed oil. All he was going to say before was that eighteen months ago Dr. Brown could not have done so. [Laughter.]

During the last month, when Dr. Brown analyzed the sample, he found there was a considerable proportion of cottonseed oil, and also of stearine, either mutton or beef fat. Lard adulterated in that way, Dr. Brown would show, was a very inferior article indeed, was not of the same value, and was not as useful for the purposes for which genuine lard is used. The cottonseed oil produced in one season in the United States amounted, he believed, to 180 or 200 million pounds weight. Of that a very large percentage, pretty nearly half, was used by manufacturers of what was called refined lard, but which the prosecution suggested was adulterated lard. The price of cottonseed oil was only about 22s. 6d. a hundredweight, and the price of beef fat only 30s. a hundredweight, whereas the price of pure lard was 42s. 6d. a hundredweight. Therefore it would be seen that when such large quantities of cottonseed oil were used, enormous profits resulted to the manufacturer, who could substitute for the more valuable article a cheaper one. Every week there came into Liverpool £20,000 to £30,000 worth of lard, and the whole of that he presumed was used in the preparation of food. It was consequently a serious matter for the consumer, as well as a matter of considerable importance to the fair traders in lard, who offered for sale the genuine article.

Inspector Baker deposed to visiting the defendants' warehouse, and purchasing a bucket of lard on which was the name of Fairbank & Co.

Mr. Pickford did not cross-examine.

Dr. Campbell Brown, examined by Mr. Marks, stated that on the 15th of May he received the bucket of lard from the last witness and analyzed it. The result of his analysis was that he found the lard to contain a very large quantity of cottonseed oil and beef or mutton fat. He estimated the total quantity as considerably more than 40 per cent. He really believed the quantity was more than 50 per cent., but he was certain that it was more than 40 per cent.

Cross-examined by Mr. PICKFORD:

Q. I suppose you mean the cottonseed oil and the beef or mutton fat together made 40 per cent.?—A. Yes.

Q. You don't distinguish the one from the other?—A. I have not done so.

Q. Can you do so?—A. I can't tell the precise quantity of beef fat.

Q. Then, I assume, if you can't determine the quantity of beef fat, that you can't determine the quantity of cottonseed oil?—A. I am quite certain there was more than 30 per cent. of cottonseed oil, but I can't determine more. I can't estimate exactly the quantity of beef fat, and therefore I can't tell precisely the quantity of cottonseed oil over 30 per cent.

Q. Does not your test tell you anything about the beef-fat stearine?—A. It tells a good deal about it, but not the precise quantity.

Q. How do your tests show the presence of these things?—A. I think you would require to attend a course of lectures on chemistry before you do it. [Laughter.]

Mr. RAFFLES. I think we had better not have that.

By Mr. PICKFORD:

Q. What kind of tests do you use?—A. I put the whole thing through seven or eight processes and then argue the thing out. I have no individual test.

Q. Supposing there was no cottonseed oil at all?—A. Yes, but there is. [Laughter.]

Q. Supposing there was no cottonseed oil, but beef fat only added to the lard, could you distinguish that fat from the other fat?—A. I can distinguish beef fat from hog's fat.

Q. These are, as a matter of fact, new tests?—A. They are new applications of old knowledge.

Q. I mean that you could not until quite recently distinguish cottonseed oil or beef fat in the lard?—A. I was quite certain about the cottonseed oil fifteen months ago, but I did not see my way to get out the quantity sufficiently for judicial purposes. Four or five years ago I knew about a certain quantity of beef fat. I don't want to give my results now, for two reasons. One is that I am getting the quantity less every week, and the other is I don't want to let the makers of lard know how little they can put in without detection. [Laughter.]

Mr. Pickford, for the defense, said his worship would probably have divined from the cross-examination that he was not going to deny the presence of cottonseed oil in this refined lard, but he was going to say that "refined lard" was a perfectly well-known trade term, and everybody was aware that it was a compound of fats and not real hog fat. If that was proved, and the effect of the notice on the barrel that it was refined lard meant what he said, then he should bring the case within the case which was decided without mustard, in which a man asked for mustard, and on the packet was a notice that the contents were not pure mustard, but compound mustard. He did not think he was wrong in trying to stop what seemed to him to be irrelevant statements—of which a number had been made—about the comparative prices of the ingredients.

Mr. MARKS. I can prove my statement if my friend wants.

Mr. PICKFORD. A great number of these statements are absolutely inaccurate.

Mr. MARKS. I think it is only fair to myself to say what I base my statements upon.

Mr. RAFFLES. I have nothing to do with it.

Mr. MARKS. I was basing my statement upon the general broker's trade circular of the 15th of last month.

Mr. RAFFLES. Don't let us talk about it.

Mr. PICKFORD. No, sir; I don't want to. But if I allow it to pass without contradiction it would no doubt be stated in the papers that it was admitted as a fact, and I don't admit it. I do admit that this refined lard is sold at about the same price as pure lard, and I admit that as being important for showing that the people who use a very large quantity of this stuff in this country willingly pay the same price, and have no complaint to make of the stuff.

Mr. Pickford was proceeding to make remarks upon the summons when—

Mr. RAFFLES said: The only question I have to deal with is the adulteration.

Mr. PICKFORD. But is it adulterated?

Mr. RAFFLES. On Dr. Campbell Brown's evidence it is adulteration.

Mr. PICKFORD. I say it is not adulteration. It is sold as refined lard, and I can call evidence—

Mr. RAFFLES. I can't go into that.

Mr. PICKFORD. If you say you can't hear evidence on that I can't say any more.

Mr. RAFFLES. I can't.

Mr. PICKFORD. But supposing that the meaning of the trade term "refined lard" is not pure hog's lard, then it is a compound and not adulterated.

Mr. RAFFLES. I can't go into any special meaning which is attached by the trade to refined lard.

Mr. PICKFORD. If the words "refined lard" do mean a compound of fats, it means that everybody who buys the stuff, that the purchaser is not getting an article of a different nature, quality, and substance to that demanded—

Mr. RAFFLES. Well, of course, this case will go elsewhere, whatever my decision is.

Mr. PICKFORD. Then I understand that you reject any evidence on my point?

Mr. RAFFLES. Yes.

Mr. Pickford said he ought perhaps to state that he intended to show that refined lard was a well-known term, meaning a compound of fats and oil, and was not con-

fined only to the American market, but the English refiners, he believed, used some cottonseed oil. Refined lard, whether American or English, was never pure hog's fat, and everybody knew it.

Mr. RAFFLES. Dr. Campbell Brown tells me this is adulterated, and that is quite sufficient for me. He will take the case elsewhere, I suppose.

Mr. PICKFORD. It is very likely, but I can't say anything one way or the other.

Mr. RAFFLES. It is a very serious question, and one which ought to be dealt with seriously. I shall inflict a fine of £5 and costs.

Messrs. Pelling, Stanley & Co., Victoria street, were summoned, and Mr. Mulholland appeared for the defense.

Mr. RAFFLES. What is the difference between this case and the last?

Mr. MARKS. An important one, in one respect, inasmuch as the lard is the same manufacture, but is from the other great refiner's. This also is called refined lard, but whether the defense means to rely upon that or not I don't know.

Inspector Baker gave evidence of the purchase of the lard from the defendants, and stated that when he offered to leave a sample with them they declined to receive it. He then took the lard to Dr. Campbell Brown.

Dr. Campbell Brown said he analyzed the lard in question and found a very large quantity of cottonseed oil and fat extracted from beef or mutton. He estimated the total quantity, approximately, at 40 per cent. The case was not so bad as the last.

Mr. Mulholland said that the witness had analyzed two samples of lard of the same brand, and had given different figures. He therefore applied for the case to be adjourned for the lard to be sent to Somerset House for analysis.

Mr. Raffles said he should decide the case himself.

In cross-examination Dr. Campbell Brown said he believed that of late years the oil formerly taken out of hog's fat to make it harder and more fit for carriage was not now salable because of the introduction of mineral oils for lubrication.

Mr. MULHOLLAND. And therefore it becomes commercially more important to put hardening stuff into the lard than to take out the oil?

The WITNESS. It becomes important to the manufacturers, but it is not right.

Mr. MULHOLLAND. You are not the analyst of right and wrong, but only of fat and stearine. [Laughter.]

Mr. Mulholland submitted that the case came within the first subsection of the act, namely, that these substances were not introduced for any fraudulent purpose, but for the purpose of hardening the lard and making it merchantable.

Mr. RAFFLES. It can not be for the purpose of bringing it over. I am against you there.

Mr. Mulholland said he also took the same point as Mr. Pickford, and as his worship was against them there he supposed he would grant them a case on that as well as on other points if necessary.

Mr. RAFFLES. I will give you any means you may require for testing my decision. It is very desirable this matter should be settled. I shall inflict the same penalty of £5 and costs.

Messrs. J. & T. Edwards, provision dealers, Whitechapel, were summoned for a similar offense.

Mr. Edwards, a member of the firm, appeared and stated that he bought the lard as pure lard.

Mr. MARKS. If you did I should sue the manufacturers for the amount of the fine and costs.

The invoice for the lard was handed to his worship, who, however, said it was no warranty of purity.

Mr. Edwards said he bought the lard as pure, and consequently he thought the fine ought to be less than in the other cases.

Mr. Raffles said he should inflict the same fine, £5 and costs.

SUMMARY.

I have endeavored to set forth in the preceding pages our present knowledge concerning the constitution of pure lard and its adulterations. The question of the wholesomeness or unwholesomeness of the various ingredients has not been raised in these investigations. It is hardly necessary to call attention, however, to the fact that the stearines and cotton oils used in the manufacture of adulterated lard are, so far as known, perfectly wholesome and innocuous. There is every reason to believe these are fully as free from deleterious effects upon the system as hog grease itself.

A more serious question which is presented is the effect of selling adulterated lard as pure lard or refined lard. To do this is a fraud upon the consumer. Although it has been claimed by the large manufacturers of refined lard that the term refined is a trade-mark whose meaning is perfectly well known by seller and purchaser, yet it can not be denied that the meaning of the word refined in the above sense is generally unknown to the consumer. The idea conveyed to the ordinary consumer by the word refined would be an article of superior purity for which he would possibly be willing to pay an increased price. It is gratifying to know that since the investigations recorded above were commenced the largest manufacturers of compound lard in this country have decided to abandon the use of the term refined and to sell their lards as compound lard or lard compounds, and, in cases where no hog grease at all enters the composition of the article, to place it upon the market as cottolene or cotton-seed oil product.

In the cases before English courts cited above it is seen that the word refined does not convey to the judicial mind the idea which is claimed for it as a trade-mark, and hence the wisdom of the manufacturers in changing the labeling of their wares is at once manifest.

The extensive adulteration of American lards has afforded grounds to foreign countries for prohibiting importation of our production or of levying upon it a heavy duty. By requiring all food products made in this country to be labelled and sold under their true name we could secure for our products immunity from any such exclusion from foreign countries as is mentioned above. The right of foreign countries to levy an import duty on our products is one which we would in no measure seek to abridge; yet by the recognized purity of our exported food articles we should see that they secure a proper entrance into foreign countries. These remarks are not alone applicable to lard and its adulterations, but to all kinds of food products, whether they are to be consumed at home or abroad.

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U.S. DEPARTMENT OF AGRICULTURE.

DIVISION OF CHEMISTRY.

BULLETIN

No. 13.

FOODS

AND

FOOD ADULTERANTS.

INVESTIGATIONS MADE UNDER DIRECTION OF

Dr. H. W. WILEY, CHIEF CHEMIST.

PART FIFTH:

BAKING POWDERS.

BY

C. A. CRAMPTON, ASSISTANT CHEMIST.

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PREFATORY NOTE.

WASHINGTON, D. C., *August 17, 1889.*

SIR: I submit herewith for your examination and approval Part Five of Bulletin No. 13 on the adulteration of food. The present part consists of an investigation of baking powders and a résumé of our present knowledge of the subject.

In these investigations we have used every endeavor to avoid error and bias. No particular powder has been favored at the expense of any other one. Our samples have been purchased in the open market and we have had them to represent as fairly as possible the character of the goods sold.

In such an investigation it is not possible to get results which will please every dealer and manufacturer, and we may therefore expect that many of our data will be distorted or denied by interested parties. A more serious embarrassment may also confront us, and that is the use of isolated portions of this report for advertising purposes.

The public official who lends the name and authority of his office for advertising purposes has little regard for either, and less for the proprieties of his position. He has, however, no longer control of the data of his analyses when they have once been published by the proper authority.

It would be well, in view of such facts, if the use of such matter for advertising purposes could be absolutely forbidden. In the present case I would like to emphasize the statement that any data or statements in the present Bulletin which may be paraded by advertisers in praise of their wares would show a discrimination wholly unauthorized by the spirit and scope of this work.

Respectfully,

H. W. WILEY,
Chemist.

Hon. J. M. RUSK,
Secretary of Agriculture.

LETTER OF SUBMITTAL.

SIR: I have the honor to submit herewith Part Fifth of Bulletin No. 13, containing the results of an investigation into the character and composition of baking powders, in continuation of the work on food adulteration. My absence in connection with the sugar experiments has greatly delayed the completion of the work and the preparation of this report.

My thanks are due to Mr. K. P. McElroy and Mr. T. C. Trescot for the intelligent assistance rendered by them in the performance of the analytical work.

Respectfully,

C. A. CRAMPTON,
Assistant Chemist.

Dr. H. W. WILEY,
Chief Chemist.

AERATION OF BREAD.

When bread is made by simply mixing flour with water and baking the dough, the result is a hard, tough, compact mass, "the unleavened bread" of the Scriptures. The use of yeast to "leaven" the dough is doubtless almost as old as the art of baking itself. Both kinds of bread are mentioned in Mosaic history, and its use was known in Egypt and in Greece at very early periods. Nothing has ever been found that could equal the action of yeast as a leavening agent. Carbonic-acid gas is generated by fermentation from the carbohydrates already existing in the bread, so that no foreign materials are introduced into it. The disengagement of the gas takes place slowly, so that it has its full effect in the lightening of the dough. This is an objection to its use, of course, when quick raising is desirable, and it is this slow action of yeast which has been the chief cause of the introduction of a chemical aerating agent.

The method of aeration invented by Dr. Danglish, in England, in March, 1859, approximates more closely the action of yeast than any other method in so far as it introduces no permanent foreign substance into the bread. In this method water which has been previously charged with carbonic dioxide is used in making up the dough, the operation being performed in a closed vessel, under pressure. As soon as the dough is taken from this vessel it immediately rises, from the expansion of the gas contained in it. The method has been modified by using instead of water a weak wort, made by mashing malt and flour, and allowing fermentation to set in. This acid liquid absorbs the gas more readily, and perhaps has some slight effect on the albuminoids, the peptonization of which constitutes an advantage of yeast-raised bread over that made by this method, in which the aeration is purely a mechanical operation. Thus the bread made by this process is somewhat tasteless, the flavors produced by fermentation within the bread being wanting. On the other hand, there is no danger of the improper fermentations which sometimes occur, and the process is especially adapted to flours which would be apt to undergo such changes when fermented. Jago¹ says with reference to it:

Working with flours that are weak or damp or even bordering on the verge of unsoundness, it is still possible to produce a loaf that should be wholesome and palatable, certainly superior to many sodden and sour loaves one sees made from low-quality flours fermented in the ordinary manner. In thus stating that it is possible

¹ Chemistry of Wheat, Flour, and Bread, and Technology of Bread-making. London. 1886.

to treat flours of inferior quality by this aerating method, the author wishes specially to carefully avoid giving the impression that it is the habit of those companies which work Daughlish's method to make use of only the lower qualities of flour; he has never had any reason whatever for supposing such to be the case.

This method is in operation in all the larger cities of Great Britain, but I have no knowledge of its being used in this country.

CHEMICAL AERATING AGENTS.

The necessity of sometimes having bread preparations raised quickly for immediate baking led to the use of chemical agents for this purpose. In all of these the expansive gas is the same as where yeast is used, but instead of its being derived from the constituents of the flour, it is obtained by the decomposition of a carbonate which is introduced, together with an acid constituent to act upon it, directly into the flour. When water is added to make the dough the chemicals are dissolved, the reaction occurs, and the carbonic acid is set free, while the salt resulting from the combination of the acid with the alkaline base of the carbonate remains in the bread and is eaten with it. Many suppose, and this idea is fostered by baking-powder manufacturers, that nothing remains in the bread, that everything is driven off during the baking. This is entirely erroneous, of course, and the residue necessarily left in the bread by baking-chemicals constitutes an objection to their use, and its amount and character determine to a large extent the healthfulness of the combination used. The essential elements of such a combination are, first, a carbonate or bicarbonate which contains the gas combined with an alkaline base, and, second, an acid constituent capable of uniting with the base in the carbonate and thus liberating the carbonic-acid gas. For the alkaline constituent bicarbonate of soda, "baking-soda," is almost exclusively employed—bicarbonate of ammonia much less. For the acid constituent, however, there is great diversity in the agents used. When the housewife mixes sour milk with baking-soda to "raise" her griddle-cakes, she makes use of the free lactic acid of the former as the acid constituent of her chemical aerating agent. When she uses "cream of tartar" or acid tartrate of potassium with soda, she uses the free tartaric acid of the former as an acid constituent, and this is the same combination that is used in one class of the baking-powders sold in the market. In fact, the entire line of such powders now sold is practically the outcome of the old-time operation of domestic chemistry, mixing "saleratus" and "cream of tartar" to aerate rolls, muffins, pancakes, and such bread preparations, which were to be baked immediately after mixing, and could not well wait for the slow operation of yeast. They consist of an acid and an alkaline constituent in about the proper proportions for combination, and in a dry state, together with various proportions of a dry inert material, such as starch, added to prevent action between the chemicals themselves, so that the preparation may be kept indefinitely.

CONSUMPTION OF BAKING-POWDERS.

The quantity of the different chemical preparations made and consumed under the name of "baking-powders," "yeast-powders," etc., in the United States can not be stated with any degree of accuracy; neither the Statistical Division of this Department nor the Bureau of Statistics of the Treasury was able to give any information whatever upon this subject. Mr. F. N. Barrett, editor of the "American Grocer," advised me that the New York Tartar Company would probably be best able to give something of an idea, at least, of the amount produced. A letter of inquiry sent to this firm elicited the following response:

DEAR SIR: Your note of inquiry of the 22d instant was received in due course of mail. We have delayed reply thereto because of the difficulty of securing with any degree of reliability the information you seek. We believe that no one can give a correct estimate of the quantity of baking-powder annually consumed in the United States, but we are led to conclude from rather careful consideration that it amounts to between 50,000,000 and 75,000,000 pounds. Of this quantity probably two-thirds is made from cream of tartar, and the residue from phosphate and alum.

Very respectfully,

NEW YORK TARTAR COMPANY.

This would seem rather a high estimate, implying as it does an annual average consumption of a pound each by every man, woman, and child in the country. Probably few persons would suppose that it reached such a figure. Taking the price per pound at 50 cents, which is about the maximum retail price charged the consumer, together with the lower of the two figures given above, we would have \$25,000,000 as the amount annually paid by consumers for this one article.

Granting that the above is somewhat of an overestimate, there can be little doubt that no other article which enters into the composition of food-stuffs, and which is not of itself a nutrient, is the subject of so great an expenditure.

The consumption of baking-powders does not seem to have become so extensive in Europe as in the United States, judging from the very small amount of attention bestowed upon the subject in works on food. Jago¹ makes but slight mention of their use. Doubtless the American people eat more largely of preparations of breadstuffs which are baked quickly, such as rolls, buns, etc.

In view of the large quantity of these preparations now consumed, and a lack of knowledge amongst most people concerning their composition and the chemical reactions that occur in their use, I have thought it proper to give a somewhat detailed exposition of the principles involved, and to endeavor to explain, even to the non-scientific reader, how these powders are made, and how they act.

¹ Op. cit.

RECENT INVESTIGATIONS.

Two important studies of the composition and character of baking-powders have been made recently, one under the direction of the Ohio Dairy and Food Commission, and the other by the Dairy Commissioner of the State of New Jersey.¹ Work done in this way, which has the authority and weight of official sanction, is most valuable, and I have drawn largely upon the reports above mentioned in the following pages. Many other analyses of baking-powders have been made from time to time, and several extensive investigations have been carried out upon the relative merits of different kinds of powders. In fact "baking-powder literature" is quite extensive. The active competition between makers of different brands, and the methods used by them in advertising their goods, have made readers of newspapers and magazines familiar with all sorts of parti-colored statements about baking-powders in general, and certain classes and brands in particular, and unfortunately such matter is not always confined to advertising columns. Most persons know comparatively little about baking-powders, and the general ignorance on the subject is taken advantage of and intensified by the manufacturers. The analyses and testimonials of eminent chemists frequently appear in such advertisements, and are often couched in terms that do little credit to the profession. I can make no use of such publications; the only material I can accept as trustworthy are the reports cited above, where the official character of the work done affords ample assurance that the investigators were influenced by unbiased and disinterested motives. It is the proper province of such bodies as State boards of health to make investigations of this kind, and results arrived at in this way are always entitled to credence, while the conclusions of scientific men, however expert they may be, are always open to doubt when they receive compensation from parties who are interested in having the results lean in their direction.

ADULTERATION.

There is no recognized standard for the composition of a baking-powder, either in this country or abroad. To prove from a legal point of view that a powder was adulterated, it would be necessary to show that it contained some substance injurious to health. Most of the treatises on food adulteration give but little attention to this class of

¹ While the present publication was passing through the press, I have received another official publication upon this subject, constituting Bulletin No. 10 of the Laboratory of the Inland Revenue Department, Ottawa, Canada, and prepared by A. McGill, assistant to chief analyst. I regret that it appeared too late to allow of the incorporation into the present publication of any of the results and conclusions contained in it. Most of the powders examined were of Canadian manufacture, but the leading American brands were also included, and the analyses were quite complete.

substances, which, though not of themselves articles of food, enter into the composition of food preparations. Considerable space is devoted in such works, however, to the adulteration of bakers' chemicals. If a substance is sold as cream of tartar, for instance, which either is not cream of tartar, or is sophisticated with some cheaper substance, the seller could be convicted under food-adulteration laws, but if such a fraudulent cream of tartar were incorporated into a mixture with other chemicals and the whole sold as baking-powder, no conviction could be secured. In the famous "Norfolk baking-powder case" in England, which will be alluded to further on, the powder in question contained alum, which substance bakers are not allowed by law to use in bread. Yet the prosecution was not successful because it was directed against the sale of the powder, not against the bread made from it, there being no legal standard for substances sold as baking-powder in England.

CLASSIFICATION OF BAKING-POWDERS.

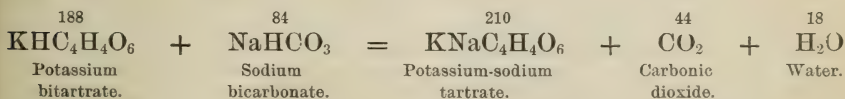
Baking-powders may be conveniently classified according to the nature of the acid constituent they contain. Three principal kinds may be recognized as follows:

- (1) Tartrate powders, in which the acid constituent is tartaric acid in some form.
- (2) Phosphate powders, in which the acid constituent is phosphoric acid.
- (3) Alum powders, in which the acid constituent is furnished by the sulphuric acid contained in some form of alum salt.

All powders sold at present will come under some one of these heads, although there are many powders which are mixtures of at least two different classes.

TARTRATE POWDERS.

The form in which tartaric acid is usually furnished in this class is bitartrate of potassium, or "cream of tartar." Sometimes free tartaric acid is used, but not often. Bitartrate, or acid tartrate of potassium is made from crude argol obtained from grape juice. It contains one atom of replaceable hydrogen, which gives it the acidity that acts upon the carbonate. The reaction takes place according to the following equation:

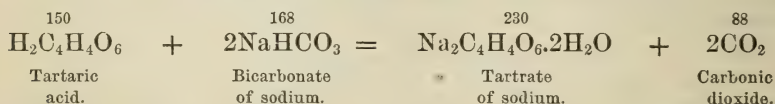


It will be seen that the products of the reaction are carbonic acid and double tartrate of potassium and sodium, the latter constituting the residue which remains in the bread. This salt is generally known as Rochelle salt, and is one of the component parts of seidlitz powders.

A seidlitz powder contains 120 grains of this salt, but the crystallized salt contains four molecules of water, and thus the actual amount of crystallized Rochelle salt formed in the baking-powder reaction is greater than the combined weight of the two salts used. That is to say, if 184 grains of bitartrate and 84 grains of bicarbonate are used in a baking there will be a residue in the dough equal to 282 grains of Rochelle salt. The directions that accompany these powders generally give two teaspoonfulls as the proper amount to use to the quart of flour; probably more is generally used. This would be at least 200 grains; deducting 20 per cent. for the starch filling we have 160 grains of the mixed bitartrate and bicarbonate, and this would form 165 grains of crystallized Rochelle salt in the loaf of bread made from the quart of flour, or 45 grains more than is contained in a seidlitz powder. The popular idea is that the chemicals used in a baking-powder mostly disappear in baking, and that the residue left is very slight. I doubt if many persons understand that when they use tartrate powders, which are considered to be the best class, or at least one of the best classes of such powders, they introduce into the breadstuff very nearly an equal weight of the active ingredient of seidlitz powders, and in a loaf of bread made from it they consume more than the equivalent of one such powder.

Yet the character of this residue is probably the least objectionable of any of those left by baking-powder. Rochelle salt is one of the mildest of the alkaline salts. The dose as a purgative is from $\frac{1}{2}$ to 1 ounce. "Given in small and repeated doses it does not purge, but is absorbed and renders the urine alkaline." (United States Dispensatory.)

Free tartaric acid, used instead of the bitartrate of potassium, would give less residue. In this case the reaction would be as follows :



Here 150 grains of tartaric acid, with 168 grains of bicarbonate of sodium, give 230 grains of residue, or 88 grains less than the combined weight of the two ingredients. As to the character of this residue little is said in regard to the physiological properties of tartrate of sodium in the books, but probably it is essentially similar to the double tartrate. The United States Dispensatory says of it (p. 1762):

This salt, in crystals, has been recommended by M. Delionx as an agreeable purgative, almost without taste, and acting with power equal to that of the sulphate of magnesium in the dose of 10 drachms [600 grains].

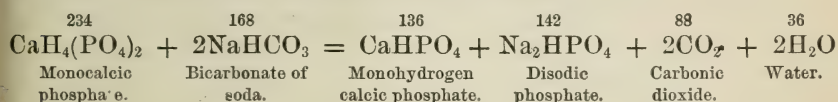
I do not know why this combination should be used so seldom by baking-powder manufacturers. The free tartaric acid is more expensive than the bitartrate, but less of it is required in proportion to the amount of bicarbonate used. The former is more soluble, and this would probably be a practical objection to its use, as it is an object in baking-powders that the gas should be liberated slowly. It would per-

haps be more difficult also to prevent action of the free acid upon the alkali, so that the powder would be more likely to deteriorate in keeping. Only one sample among those I examined was found to have been made with the free acid.

One obstacle formerly encountered in the manufacture of bitartrate powders was the difficulty of obtaining the bitartrate pure. It contained from 5 to 15 per cent. of tartrate of lime incident to the method of manufacture. This brought a large quantity of inert material into the powder and lowered its efficiency. Bitartrate can now be had 98 per cent. pure, quoted and guaranteed as such in the markets, so that there is no excuse for manufacturers to use the impure salt, which can properly be considered adulterated.

PHOSPHATE POWDERS.

The salt commonly used to furnish the phosphoric acid in this class is acid phosphate of lime, sometimes called superphosphate. The pure salt is monocalcic phosphate, $\text{CaH}_4(\text{PO}_4)_2$. It is made by the action of sulphuric acid upon ground bone, the result being an impure monocalcic phosphate with calcium sulphate. This mixture is sold as a fertilizer, as superphosphate. The salt is, of course, more or less purified for use in baking-powders, but the sulphate of lime is very difficult to get rid of entirely, and most phosphate powders contain considerable amounts of this impurity. The reaction which occurs when a phosphate powder is dissolved, that is the action of bicarbonate of soda upon monocalcic phosphate, is not well established, and perhaps varies somewhat with conditions. The following equation probably represents it fairly well:



Two hundred and thirty-four grains of monocalcic phosphate combined with 168 grains of bicarbonate of soda give 136 grains of monohydrogen calcic phosphate, and 142 grams of disodic phosphate. But crystallized sodic phosphate contains twelve molecules of water, and has a molecular weight of 358. So the total amount of residue from 402 grains of the powder would be 494 grains, of which 136 grains is phosphate of lime, and the rest phosphate of soda. So we see that here also the quantity of chemicals introduced into the dough is fully equal to the amount of the baking-powder used, including filling. As to the nature of this residue in phosphate powders, it would seem to be about as unobjectionable as in the tartrates. Phosphate of soda is "mildly purgative in doses of from 1 to 2 ounces" (480-960 grains), according to the United States Dispensatory. Phosphates of calcium have the general physiological effect which is ascribed to all forms of phosphoric acid, but which does not seem to be well understood.

Phosphates are administered therapeutically in some cases of defec-

tive nutrition, and especially in scrofula, rickets, phthisis, etc. On account of their being an essential constituent of animal tissues there would seem to be some ground for a preference over other forms of powders. The makers of phosphate powders claim that the use of such powders restores the phosphoric acid present in the whole grain of wheat, which is largely removed in the bran by milling processes. This claim would have more weight if there were not ample sources of phosphoric acid in other forms of food, and if the quantity introduced by a baking-powder were not much greater than is required to make up the loss in the bran, and greater than is required by the system, unless in those cases where its therapeutic use is indicated, as in some of the conditions of malnutrition given above.

Acid phosphate of soda is said to have been used in former years as a constituent of baking-powders, but appears to have been entirely superseded by the lime salt.

ALUM POWDERS.

In this class the carbonic acid is set free from the bicarbonate by the substitution of sulphuric acid, which combines with the sodium. The sulphuric acid is furnished by some one of the general class of salts known as alums, which are composed of a double sulphate of aluminium and an alkali metal. The alum is precipitated as hydrate, while that portion of the sulphuric acid which was combined with it goes to displace the carbonic acid in the bicarbonate. The alkali sulphate of the double salt remains unchanged.

The alum of commerce is either *potash alum*, $K_2Al_2(SO_4)_4 \cdot 24H_2O$, or *ammonia alum*, $(NH_4)_2Al_2(SO_4)_4 \cdot 24H_2O$, the one or the other predominating according to the relative cheapness of the alkali salt it contains. At the present time nothing but ammonia alum is met with, but at previous periods potash alum was the salt sold exclusively as "alum." Both salts are alike in general appearance and can not be distinguished apart by cursory examination.

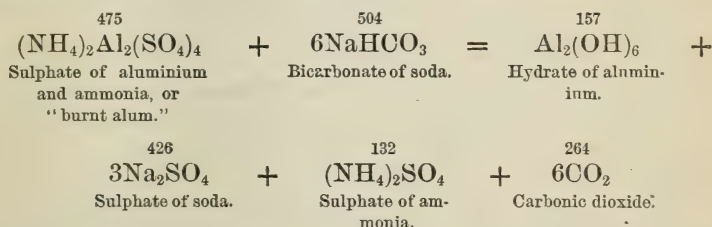
Potash alum may be made directly from some minerals, such as the "alum stone" mined in Italy, which contain all the constituents combined. Ammonia alum, however, as well as most potash alum, is made by the combination of the constituents obtained from different sources. The sulphate of alumina is obtained by the action of sulphuric acid upon pure clays, and the sulphate of ammonia from the residue of gas-works. Solutions of the two salts in proper proportions are mixed and the double salt obtained by evaporation and crystallization.

Crystallized potash or ammonia alum contains twenty-four molecules of water, nearly one-half of its weight. Part of this water is lost at as low a heat as $60^\circ C.$, and it is driven off entirely, though slowly, at $100^\circ C.$ "Burnt alum" is simply alum deprived of its water of crystallization, which is generally driven off at about $200^\circ C.$ Ammonia alum decomposes at $205^\circ C.$; potash alum at a somewhat higher temperature.

Burnt alum is somewhat hygroscopic, but dissolves more slowly in water than the crystallized salt.

I have been unable to ascertain in what condition the alum is used for compounding baking-powders. Burnt alum would seem to be the form best adapted for this purpose on account of its slow solubility. Professor Cornwall says this is the form ¹ used, but does not state how he obtained the information; and he states further that "crystallized alums may be used in connection with burnt alum to secure at first a more rapid escape of carbonic-acid gas." It is probable that the amount of drying given the alum used differs with different manufacturers, but it is not likely that the water of crystallization is entirely driven off.

The following equation shows the reaction taking place in a baking-powder made with burnt ammonia alum :



If potash alum were used the reaction would be precisely the same with the substitution of potassium for ammonia wherever it occurs in the equation, sulphate of potash being formed instead of sulphate of ammonia.

A study of the equation will show that 475 grains of burnt alum with 504 grains of bicarbonate will produce 264 grains of carbonic acid and leave a residue consisting of 426 grains of sulphate of soda, 132 grains of sulphate of ammonia, and 157 grains of hydrate of aluminium, the last named being a precipitate insoluble in water. Sulphate of soda crystallizes with ten molecules of water, so that the total weight of residue from the 979 grains of mixed chemicals would be 1,255 grains. If a hydrated alum is used in the powder, the proportion of residue to powder would of course be less, and the proportion of gas evolved would also be less. The character of the residue is seen to be more complex than is the case with any of the classes previously discussed, and deserves special attention. The sulphate of soda is similar to other alkali salts in its physiological action. Sulphate of ammonia is not used therapeutically, but probably has an action similar to that of other ammonia salts, such as the chloride. Professor Cornwall,² in his report, speaks as follows concerning this point :

It is possible, however, that too little attention has been paid to the presence of ammonium salts in the residues from ammonia alum powders. * * * We do know, however, that ammonia salts, in general, are much more irritating and stimulating in their action than the corresponding soda salts, or even than the potash salts. For instance, Stillé and Maisch, speaking of ammonium bromide, state that it has a

¹ Report of the Dairy Commissioner of New Jersey, 1888, p. 70. ² Op. cit., p. 77.

more acid taste and is more irritating than potassium bromide. Its unpleasant taste and irritating qualities render it less convenient for administration than the bromide of potassium.

We all know how mild a substance is chloride of sodium (common table salt); but of ammonium chloride Stillé and Maisch write: "The direct effects of doses of 5 to 20 grains of this salt, repeated at intervals of several hours, are a sense of oppression, warmth, and uneasiness in the stomach, some fullness in the head. If it is used for many days together in full doses, it disturbs the digestion, coats the tongue, and impairs the appetite." We have already seen how active a drug carbonate of ammonia is, and while, in the absence of proof, it would be rash to assert that sulphate of ammonia in five-grain doses is certainly injurious, yet there is abundant ground for further investigating its effect before asserting that it is milder in its effects than Rochelle salt. It may be that this question of the presence of ammonium salts in any considerable quantities in the residues of baking-powders deserves more attention than it has hitherto received.

It would seem from the above that there would be considerable difference between the physiological effects of potash and ammonia alums themselves. Yet the medical authorities make no such distinction. Ammonia alum is officinal in the British Pharmacopœia, and while the United States Pharmacopœia specifies potash alum, the particular form met with in trade is entirely determined by the comparative cheapness of manufacture.

The question of the relative harmfulness of these different salts in the residues of baking-powders is really one for the physiologist or hygienist to decide, not the chemist. Physiological experiments alone can decide them positively.

The consideration of the residue of hydrate of aluminium will be taken up later on.

POWDERS CONTAINING MORE THAN ONE ACID INGREDIENT.

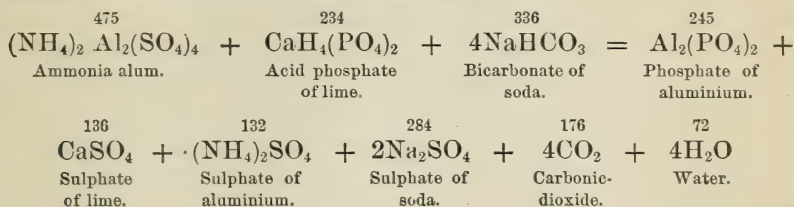
As might be expected, some powders are met with which have been made up with various proportions of different acid ingredients, and which belong therefore to more than one of the above-mentioned classes. Professor Cornwall speaks as follows concerning some of these mixed powders:

The makers of alum baking-powders sometimes add tartaric acid or bitartrate to their powders, either with or without the addition of acid phosphate of lime. This is doubtless done with the best intentions, either to secure a more rapid escape of carbonic-acid gas at the outset, or otherwise improve the powder. We have found such additions in the case of several of our samples, but the presence of tartaric acid or tartrates in alum powders is very objectionable. If added in sufficient quantity to otherwise pure alum powders, they prevent the precipitation of the insoluble hydrate of aluminium entirely when the powder is boiled with water, and they may render much of the alumina soluble in water even after the bread is baked. Without doubt it would then be readily soluble in the digestive organs, producing there the effects due to alum or any other soluble aluminium compound. With one of our samples we found that the simple water solution seemed to contain as much alumina as a nitric-acid solution. In neither of these solutions could any of the alumina be thrown down by a slight excess of ammonia water, although it was readily precipitated from the solution first rendered alkaline with caustic soda, then slightly acidified with acetic acid and boiled with excess of phosphate of soda.

A case in which the character of the powder appears to be improved by such mixing, however, is furnished by the

ALUM AND PHOSPHATE POWDERS.

This combination seems to be a favorite one with manufacturers. In fact there are now comparatively few "straight" alum powders in the market, most of the cheaper grades being made of mixtures in various proportions of the alum with acid phosphate of lime. The reaction it is intended to obtain is probably the following :



If this equation be compared with the one representing the reaction in a powder made with alum alone, on page 569, it will be seen that in the former the alum goes into the residue as phosphate instead of hydrate, and the insoluble sulphate of lime takes the place of one molecule of sulphate of soda. Otherwise the reactions are similar. This reaction will only take place, of course, when the different ingredients are mixed in just the proper proportions to produce it. A number of variations may be produced by changing the relative proportions of the different ingredients.

THE "ALUM QUESTION."

The literature upon the subject of the use of alum in baking-powders, and upon the question as to its injurious effect upon the health of those who consume the bread made from it, is already quite extensive, and if quoted entire would fill a fair-sized volume. For the benefit of those who may desire to make an exhaustive study of it, I will make reference to all of the articles bearing upon the subject that have come under my observation as follows :

Alum in baking-powder, by Prof. E. G. Patrick.—*Scientific American Supplement* No. 185, 7, p. 2940.

Report of proceedings in the Norfolk baking-powder case (first trial).—*Analyst*, 4, p. 231.

Norfolk baking-powder case (second trial).—*Ibid.*, 5, p. 21.

Editorial comment on the case.—*Ibid.*, 5, pp. 13 and 34.

On the action of alum in bread making, by J. West Knights.—*Ibid.*, 5, p. 67.

Cereals and the products and accessories of flour and bread foods, by E. G. Love, Ph. D.—*Second Annual Report State Board of Health of New York*, 1882, p. 567.

On the solubility of alumina residues from baking-powders, by Lucius Pitkin.—*Journal American Chemical Society*, 9, p. 27.

Experiments upon alum baking-powders and the effects upon digestion of the residues left therefrom in bread, by Prof. J. W. Mallet.—*Chemical News*, 58, pp. 276 and 284.

As I have previously indicated, the matter of the physiological effect of the residues left by baking powders is not properly a chemical problem. On account of the interest and importance attached to it, however, it would seem necessary to give here somewhat of a résumé of the subject without attempting to arrive at a definite conclusion, or to settle, arbitrarily, the question as to whether the sale of certain forms of powders should be prohibited.

For a proper understanding of the alum question it is necessary to explain that the use of alum in bread-making is prohibited in countries having food-adulteration laws, such as England and France. This is partly on account of its injurious effect upon the system, but principally because of its peculiar action, not yet well understood, in improving the color and appearance of the bread to which it has been added, so that a flour of inferior grade, or even partially spoiled, may be used to make bread which will look as well, to all appearances, as bread made from much better grades.

Blyth¹ speaks as follows of this use of alum in bread :

Alum is added to bad or slightly damaged flour by both the miller and the baker. Its action, according to Liebig, is to render insoluble gluten which has been made soluble by acetic or lactic acids developed in damp flour, and it hence stops the undue conversion of starch into dextrine or sugar. The influence of alum on health, in the small quantities in which it is usually added to bread, is very problematical, and rests upon theory more than observation. But notwithstanding the obscurity as to its action on the economy there can be no difference of opinion that it is a serious adulteration, and not to be permitted.

Allen² says :

Alum, or an equivalent preparation containing aluminium, is by far the most common mineral adulterant of bread, though its use has greatly decreased of late years. Its action in increasing the whiteness and apparent quality of inferior flour is unquestionable, though the cause of its influence has not been clearly ascertained. Whether there be sufficient foundation for the statements made respecting the injurious effects of alumed bread on the system is still an open question.

The following is from Hassall:³

With reference to the use of alum, Dr. Daughlish has written : " Its effect on the system is that of a topical astringent on the surface of the alimentary canal, producing constipation and deranging the process of absorption. But its action in neutralizing the efficacy of the digestive solvents is by far the most important and unquestionable. The very purpose for which it is used by the baker is the prevention of those early stages of solution which spoil the color and lightness of the bread whilst it is being prepared, and which it does most effectually ; but it does more than needed, for, whilst it prevents solution at a time that is not desirable, it also continues its effects when taken into the stomach, and the consequence is that a large portion of the gluten and other valuable constituents of the flour are never properly dissolved, but pass through the alimentary canal without affording any nourishment whatever."

The manufacturers of alum baking-powders, however, claim that the hydrate of aluminium which is left in the residue is insoluble in the

¹ Foods, Composition and Analysis, p. 168.

² Commercial Organic Analysis, 1, p. 371.

³ Food, its Adulterations, and the Methods for their Detection, p. 344.

digestive juices, and therefore does not produce the effect which is attributed to the soluble forms of alum. Aluminium hydrate is insoluble in water, but readily soluble in dilute acids, especially when freshly precipitated. When heated it gradually loses its water of hydration, but does not part with it entirely short of a very high heat. When completely dehydrated it is insoluble even in dilute acid. It never reaches this condition in baked bread, in which the temperature probably never, in the center of the loaf, at least, exceeds 100°C .

Phosphate of aluminium is somewhat less soluble in dilute acids than the hydrate. In the Norfolk case an effort was made by the prosecution to show that the soluble phosphates contained in the ash of flour combined with the alum to form phosphate of aluminium, thus rendering them insoluble in the digestive juices, and depriving the flour of an important constituent, and considerable evidence was offered by the defense to show that this was not the case. Whether the addition to alum powders of sufficient acid phosphate to combine with the aluminium present as phosphate was the result of this discussion or not I can not say, but it is certain that most of the alum powders now met with are made in this way, so that if such a prosecution were to occur to day the relative position of the parties would be reversed. It would be to the interest of the alum-powder makers to show that phosphate of aluminium is insoluble in the alimentary canal. The solubility of these compounds in water or dilute acids is, of course, a question readily answered by any chemist, but their solubility in the complex and various alimentary fluids, and under the conditions of natural digestion in the human body, is quite another matter. As might be expected, the testimony which has been published upon this point is of the most conflicting character. Professor Patrick, experimenting upon cats, found little or no solution of hydrate of aluminium. Professor Pitkin, experimenting with gastric juice obtained from a dog, found some solution, although he used phosphoric acid in his powder. Professor Mallet, using an artificial gastric juice, found some solution to occur, even with the phosphate, and considerably more with the hydrate. It is not difficult to find reasons for such disagreement in results, for, besides the various character of the solvents used and the different conditions prevailing, it is easy to see that even if the hydrate and phosphate of aluminium were themselves entirely insoluble, more or less aluminium would escape the reaction, either from imperfect mixing of the powder in the dough or from improper proportioning of the different ingredients in the powder itself, so that it would go into the residue in the form of the original salt. With powders specially prepared, on the other hand, and very carefully mixed, and kneaded up thoroughly with the dough, it might be possible to find but a very little dissolved in the digestive fluids under certain conditions, even though the salts formed were slightly soluble in such fluids.

From the various evidence that has been produced on both sides of the question, I think the following conclusions may be safely drawn:

(1) That form of alum powder in which sufficient phosphate is added to combine with all the aluminium present is a better form, and less apt to bring alum into the system than where alum alone is used.

(2) It must be expected that small quantities, at least, of alum will be absorbed by the digestive fluids where any form of powder containing it is used.

(3) Whether the absorption of small quantities of alum into the human system would be productive of serious effects is still an open question, and one that careful physiological experiment alone can decide.

As the experiments made by Professor Mallet are the most recent on this subject, I quote here his conclusions. I may say that most of those based upon purely chemical work I can indorse, having confirmed many in my own work, but I think the evidence furnished by his physiological work is hardly sufficient to justify his conclusion as to the harmfulness of such powders.

GENERAL SUMMARY OF THE CONCLUSIONS REACHED.¹

The main points which seem to be established by the experiments under discussion are, briefly stated, the following:

(a) The greater part of the alum baking-powders in the American market are made with alum, the acid phosphate of calcium, bicarbonate of sodium, and starch.

(b) These powders, as found in retail trade, give off very different proportions of carbonic-acid gas, and therefore require to be used in different proportion with the same quantity of flour, some of the inferior powders in largely increased amount to produce the requisite porosity in bread.

(c) In these powders there is generally present an excess of the alkaline ingredient, but this excess varies in amount, and there is sometimes found on the contrary an excess of acid material.

(d) On moistening with water these powders, even when containing an excess of alkaline material, yield small quantities of aluminium and calcium in a soluble condition.

(e) As a consequence of the common employment of calcium-acid phosphate along with alum in the manufacture of baking-powders, these, after use in bread-making, leave, at any rate, most of their aluminium in the form of phosphate. When alum alone is used the phosphate is replaced by hydroxide.

(f) The temperature to which the interior of bread is exposed in baking does not exceed 212° F.

(g) At the temperature of 212° F. neither the "water of combination" of aluminium hydroxide nor the whole of the associated water of either this or the phosphate is removed in baking bread containing these substances as residues from baking-powder.

(h) In doses not very greatly exceeding such quantities as may be derived from bread as commonly used, aluminium hydroxide and phosphate produce, or produced in experiments upon myself, an inhibitory effect upon gastric digestion.

(i) This effect is probably a consequence of the fact that a part of the aluminium unites with the acid of the gastric juice and is taken up into solution, while at the same time the remainder of the aluminium hydroxide or phosphate throws down in insoluble form the organic substance constituting the peptic ferment.

¹ Chemical News, 58, 276; also published in pamphlet form.

(k) Partial precipitation in insoluble form of some of the organic matter of food may probably also be brought about by the presence of the aluminium compounds in question.

(l) From the general nature of the results obtained, the conclusion may fairly be deduced that, not only alum itself, but the residues which its use in baking-powder leaves in bread, can not be viewed as harmless, but must be ranked as objectionable, and should be avoided when the object aimed at is the production of wholesome bread.

COMPARISON OF THE DIFFERENT CLASSES OF POWDERS IN RESPECT TO THEIR RELATIVE AERATING STRENGTH AND THE AMOUNT OF RESIDUE LEFT BY EACH.

The following comparison of the different powders described may prove interesting. It is assumed, of course, that the ingredients are combined in exactly the proper proportions, and that all the chemicals used are of full purity and strength:

Powders.	Carbonic-acid gas.	Total residue of the weight of chemicals used.
	<i>Per cent.</i>	<i>Per cent.</i>
Tartrate	16	104
Phosphate	22	123
Alum	27	128
Alum and phosphate	17	111

From this it will be seen that a tartrate powder, theoretically, gives the lowest percentage of carbonic-acid gas in proportion to the weight of chemicals used in its composition, together with the least weight of residue; and a straight alum powder gives the highest proportion of gas, with the greatest weight of residue. It is assumed that burnt alum is used in both the alum and the alum and phosphate powders. The residues are calculated to hydrated salts in all cases. No account is made of inert "filling," as that would be the same in each case. It should of course be remembered that in the above calculation the *total weight* of residue is reckoned in each case without regard to solubility or relative effect upon the system of the various salts formed. This has been sufficiently discussed under the different classes.

CARBONATE OF AMMONIA.

This salt is used to some extent as an ingredient in baking-powders. It is also often used alone by bakers as a chemical aerating agent. It does not necessarily require an acid to set free its gases, being volatilized without decomposition simply in heating. The commercial salt, familiar to everybody as "smelling-salts," or *sal volatile*, is obtained by subliming a mixture of two parts of chalk and one part of sal ammoniac

or sulphate of ammonia. The salt is then resublimed with the addition of some water, and a white semi-transparent mass is obtained, which has a strongly ammoniacal smell, and a pungent, caustic taste. It has the composition $N_3H_{11}C_2O_5$, and consists of a compound of hydrogen ammonium carbonate with ammonium carbonate, $H(NH_4)CO_3 + NH_4CO_2NH_2$. "When heated the salt is wholly dissipated, without charring; if the aqueous solution is heated to near $47^\circ C$. it begins to lose carbonic-acid gas, and at 88° it begins to give off vapor of ammonia." (United States Pharmacopœia.) The question of the propriety of the use of this salt in baking does not seem to have received a great deal of attention, and opinions differ. Hassall¹ says of it:

* * * Of these, by far the best is carbonate of ammonia; this is a volatile salt, and its great advantage is that it is entirely or almost entirely dissipated by the heat employed in the preparation of the bread; and thus the necessary effect is produced without risk of injurious results ensuing.

This would doubtless hold good if it were quite certain that the salt is *entirely* driven off by the baking of the bread, for it is a very active therapeutic agent, acting as a corrosive poison when taken in sufficient amount. The ordinary dose is five grains. Doubtless in the small quantities used in baking-powders, and in the presence of other chemicals, there is little danger of its being left in the bread undecomposed, but the advisability of its use alone as an aerating agent is open to grave doubt.

COMPOSITION OF BAKING-POWDERS AS FOUND IN THE MARKET.

The following analyses and discussion, by Prof. H. A. Weber, form a part of the Annual Report of the Ohio State Dairy and Food Commissioner for 1887:

BAKING-POWDERS.

Much complaint has been made to this commission of the character of the baking-powders of commerce. It was believed by many that there was a great deal of adulteration and impurity in the ordinary baking-powders used by our people, and that the public health was seriously affected thereby. Recognizing the importance of this matter to the health and domestic economy of the people of the State, I gave public notice of my purpose to investigate the purity and healthfulness of the various brands of the baking-powders of commerce. I sought all possible information on this subject, and collected and submitted to analysis by the State chemist thirty brands of baking-powders, such as I found on sale in many sections of the State. In September the result of these investigations and analyses was published in an official circular for the benefit of the consumers of this class of goods. There was no thought, wish, or purpose upon the part of this commission to aid or to defeat the enterprise of any manufacturer of these goods. Indeed, we had not any possible intimation as to what the analysis would show in any particular brand until the work was accomplished. There was simply the impartial purpose to inform the public as to the chemical composition of the several brands sold by the trade throughout the State, so that with the knowledge of the facts they might not claim that they were being defrauded or imposed upon, but be able intelligently to choose the goods they deemed most health-

¹ Page 345.

ful and desirable. This circular with its analysis has attracted so much attention throughout the State and country and is of such significance as to demand a place in this report, and it is therefore given here in full.

CIRCULAR NO. 6.—BAKING-POWDERS.

This commission has been for some months investigating the baking-powders of commerce most generally used and sold in this State. And we herein submit to the people of the State the result of that investigation.

We have analyzed thirty brands of baking-powder, seeking those brands which were apparently most generally sold throughout the State. We submit the result of these analyses to the people who are the consumers of such goods that they may know the true chemical character of these several varieties.

It is generally supposed that there is a vast deal of "adulteration" in baking-powder, but since there is at law no standard of excellence or purity in baking-powder, it is difficult to say what is an adulteration, unless it be an unhealthful ingredient.

As a matter of fact, any powdered composition that is healthful and which in solution in moist dough will generate carbonic-acid gas and "raise" bread, or cause it to be porous and light, may be properly called a baking-powder. And accordingly we find very many varieties or brands of baking-powders on the market made from widely-different materials.

The best baking-powder is, of course, that in which (the ingredients being healthful) the largest amount of carbonic gas is generated to the spoonful of powder, and the least amount and least hurtful character of the resultant salt remains in the bread.

For an intelligent view of the whole field we classify these varieties into three general divisions. In each of these the active agents of the compound are kept dry, and thus free from fermentation in the package, by the use of a given per cent. of starch, wheat flour, or rice flour. These are used simply as dry filling to keep the chemical agents from acting upon each other in the package.

In this classification we have—

- (1) Cream of tartar baking-powders.
- (2) Phosphate baking-powders.
- (3) Alum baking-powders.

The chemical formula and the percentages of the active agents vary with each brand. But generally stated we have in the

FIRST CLASS.

Cream of tartar, Bicarbonate of soda, Starch or flour,	}	Changed by chemical action in the dough to the double salt of tartrate of potassium and sodium, and carbonic-acid gas.
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The cream of tartar and bicarbonate of soda, dissolved by the water or moisture in the dough, unite chemically and form in the bread the double salt of tartrate of potassium and sodium, and carbonic-acid gas, the latter escaping in the baking heat.

SECOND CLASS.

Acid calcium phosphate, Bicarbonate of soda, Starch or flour,	}	Changed by chemical action into calcium phosphate, sodium phosphate, and carbonic-acid gas.
---------------------------------------------------------------------	---	------------------------------------------------------------------------------------------------

THIRD CLASS.

Ammonium alum, Bicarbonate of soda, Starch or flour,	}	Changed by chemical action into hydrate of aluminium, so- dium sulphate, ammonium sulphate, and carbonic-acid gas.
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In some brands of the cream of tartar baking-powder a small per cent. of carbonate of ammonia is used; but this is considered to be too small an amount to be hurtful. There is a prevalent belief created by the erroneous statement of manufacturers, that the salts from which carbonic-acid gas is generated pass off in the form of escaping gas, scarcely leaving a trace of their presence in the bread. But this is not true. These resultant salts formed by the chemical action in the dough remain in the bread, while the gas generated by such chemical action, and which is but a small per cent. of the whole, alone passes off in the process of baking.

From this fact many persons condemn the entire class of alum baking-powders as being unhealthful. Pure alum is undoubtedly a hurtful salt, and the resultant salts

from its combination with soda can scarcely be less hurtful. And yet this is a question about which "doctors disagree;" any number of conflicting opinions and certificates can be had from eminent chemists on either side of this question.

The official investigation of this class of baking-powders made in England to test their healthfulness resulted in their favor. But this conclusion rested upon the statement of chemists that the resultant salt of hydrate of aluminium remaining in the bread was insoluble, and hence un hurtful when taken into the stomach. But some of the ablest chemists of this country declare that hydrate of aluminium is quite soluble, and hence is as hurtful as the alum in other forms. So that the question is yet an open one to be determined by further careful scientific investigation.

As to the general healthfulness of cream of tartar and phosphate baking-powders when properly used, there is but little difference of opinion; but an intelligent knowledge of their strength and freshness and of the manner and rapidity of the chemical combinations in the process of bread-making is necessary to the baker in order to insure good bread.

These thirty brands were analyzed very carefully by Prof. H. A. Weber, State chemist at Columbus, Ohio, and are such as are generally sold throughout the State. The condition of some of these brands was not such as to show them at their best advantage, since some were old while others were fresh. But since they were bought in the open market without discrimination, they fairly present what the consumer must buy.

S. H. HURST,

Ohio Dairy and Food Commissioner.

General S. H. HURST,

Ohio Dairy and Food Commissioner :

SIR: The following is a complete report of the analyses of baking-powders received June 3 and July 7, 1887:

The list, as will be seen from the analyses, includes three kinds of baking-powders, in which the acid principle is respectively cream of tartar, acid phosphate of calcium, and alum.

The carbonic acid evolved with water was determined with great care, and from this the amount of bicarbonate of soda and the acid principle was calculated according to well-known reactions.

The starch, or as in some cases the flour, was determined directly, due allowance being made in case of the alum powders for the loss of water of the aluminium hydroxide in the dried residue upon ignition.

Accidental impurities of commercial drugs were not taken into account, as they would be very small and unimportant in case of the alum powder, while in the cream of tartar powder the ingredients used were found in the course of analysis to be practically pure.

The excess of bicarbonate of soda or of the acid principle was determined volumetrically and added, as the case might be, to the results obtained by calculation.

Respectfully submitted.

H. A. WEBER,

Chemist.

ANALYSES OF BAKING-POWDERS.

CREAM OF TARTAR BAKING-POWDERS.

1. *Royal.*

Carbonic-acid gas, 11.80 per cent.

Bicarbonate of soda	25.21
Cream of tartar	50.44
Starch	17.10
Tartrate of potassium and sodium, moisture, etc	7.25
	<hr/> 100.00

This powder contained a small percentage of ammonium carbonate, which was calculated as bicarbonate of soda above.

2. *Dr. Price's.*

Carbonic-acid gas, 10.50 per cent.

Bicarbonate of soda	21.14
Cream of tartar	44.90
Starch	21.30
Tartrate of potassium and sodium, moisture, etc	12.66
	<hr/> 100.00

3. *Pearson's.*

Carbonic-acid gas, 11.60 per cent.	
Bicarbonate of soda	23.24
Cream of tartar	49.57
Starch	12.80
Tartrate of potassium and sodium, moisture, etc	14.39
	<hr/>
	100.00

This sample contained ammonium carbonate.

4. *Cleveland's.*

Carbonic-acid gas, 12.80 per cent.	
Bicarbonate of soda	26.12
Cream of tartar	54.70
Starch	9.00
Tartrate of potassium and sodium, moisture, etc	10.18
	<hr/>
	100.00

5. *Snow Drift.*

Carbonic-acid gas, 10.60 per cent.	
Bicarbonate of soda	20.24
Cream of tartar	48.62
Starch	13.60
Tartrate of potassium and sodium, moisture, etc	17.54
	<hr/>
	100.00

6. *Upper Ten.*

Carbonic-acid gas, 11.30 per cent.	
Bicarbonate of soda	21.57
Cream of tartar	48.31
Starch	20.90
Tartrate of potassium and sodium, moisture, etc	9.22
	<hr/>
	100.00

7. *De Land's.*

Carbonic-acid gas, 10.00 per cent.	
Bicarbonate of soda	19.09
Cream of tartar	48.39
Starch	0.00
Tartrate of potassium and sodium, moisture, etc	32.52
	<hr/>
	100.00

This powder contained no filling.

8. *Sterling.*

Carbonic-acid gas, 11.00 per cent.	
Bicarbonate of soda	21.84
Cream of tartar	47.03
Starch	18.50
Tartrate of potassium and sodium, moisture, etc	12.63
	<hr/>
	100.00

PHOSPHATIC BAKING-POWDERS.

9. *Horsford's.*

Carbonic-acid gas, 13.00 per cent.	
Bicarbonate of soda	27.34
Free phosphoric acid	14.47
Starch	21.80
Insoluble ash (calcium phosphate, calcium carbonate, etc.)	19.50
Sodium phosphate, moisture, etc	16.89
	<hr/>
	100.00

10. *Wheat.*

Carbonic-acid gas, 4.00 per cent.	
Bicarbonate of soda	9.32
Free phosphoric acid	4.45
Starch	0.00
Insoluble ash (calcium phosphate, calcium carbonate, etc.)	26.90
Sodium phosphate, moisture, etc.	59.33
	<hr/>
	100.00

This sample contained no filling and was badly caked.

ALUM BAKING-POWDERS.

11. *Empire.*

Carbonic-acid gas, 5.80 per cent.	
Bicarbonate of soda	11.08
Alum	10.41
Starch	44.25
Hydrate of alumina, sodium sulphate, ammonium sulphate, moisture, etc.	34.26
	<hr/>
	100.00

12. *Gold.*

Carbonic-acid gas, 6.70 per cent.	
Bicarbonate of soda	13.63
Alum	12.03
Starch	44.00
Hydrate of alumina, sodium sulphate, ammonium sulphate, moisture, etc.	20.34
	<hr/>
	100.00

13. *Veteran.*

Carbonic-acid gas, 6.90 per cent.	
Bicarbonate of soda	14.66
Alum	12.13
Starch	49.85
Hydrate of alumina, sodium sulphate, ammonium sulphate, moisture, etc.	23.36
	<hr/>
	100.00

14. *Cook's Favorite.*

Carbonic-acid gas, 5.80 per cent.	
Bicarbonate of soda	11.92
Alum	10.41
Starch	42.75
Hydrate of alumina, sodium sulphate, ammonium sulphate, moisture, etc.	34.92
	<hr/>
	100.00

15. *Sunflower.*

Carbonic-acid gas, 6.30 per cent.	
Bicarbonate of soda	14.44
Alum	11.31
Starch	38.65
Hydrate of alumina, sodium sulphate, ammonium sulphate, moisture, etc.	35.60
	<hr/>
	100.00

16. *Kenton.*

Carbonic-acid gas, 6.20 per cent.	
Bicarbonate of soda	12.59
Alum	11.14
Starch	38.10
Hydrate of alumina, sodium sulphate, ammonium sulphate, moisture, etc.	38.17
	<hr/>
	100.00

17. *Patapsco.*

Carbonic-acid gas, 6 per cent.	
Bicarbonate of soda	12.30
Alum	10.77
Starch	36.85
Hydrate of alumina, sodium sulphate, ammonium sulphate, moisture, etc.	40.08
	<hr/>
	100.00

18. *Jersey.*

Carbonic-acid gas, 10.40 per cent.	
Bicarbonate of soda	20.70
Alum	19.05
Starch	44.20
Hydrate of alumina, sodium sulphate, ammonium sulphate, moisture, etc.	16.05
	<hr/>
	100.00

19. *Buckeye.*

Carbonic-acid gas, 6.90 per cent.	
Bicarbonate of soda	13.82
Alum	12.13
Starch	44.20
Hydrate of alumina, sodium sulphate, ammonium sulphate, moisture, etc.	29.85
	<hr/>
	100.00

20. *Peerless.*

Carbonic-acid gas, 7 per cent.	
Bicarbonate of soda	14.21
Alum	12.94
Starch	46.57
Hydrate of alumina, sodium sulphate, ammonium sulphate, moisture, etc.	26.28
	<hr/>
	100.00

21. *Silver Star.*

Carbonic-acid gas, 6.90 per cent.	
Bicarbonate of soda	14.66
Alum	12.13
Starch	41.33
Hydrate of alumina, sodium sulphate, ammonium sulphate, moisture, etc.	31.88
	<hr/>
	100.00

22. *Crown.*

Carbonic-acid gas, 8.40 per cent.	
Bicarbonate of soda	16.88
Alum	15.08
Starch	51.35
Hydrate of alumina, sodium sulphate, ammonium sulphate, moisture, etc.	16.69
	<hr/>
	100.00

23. *Crown* (marked "Special").

Carbonic-acid gas, 8.60 per cent.	
Bicarbonate of soda	18.10
Alum	15.44
Starch	41.37
Hydrate of alumina, sodium sulphate, ammonium sulphate, moisture, etc.	25.09
	<hr/>
	100.00

24. *One Spoon.*

Carbonic-acid gas, 5.75 per cent.	
Bicarbonate of soda	12.66
Alum	10.33
Starch	18.33
Hydrate of alumina, sodium sulphate, ammonium sulphate, moisture, etc.	58.68
	<hr/>
	100.00

25. *Wheeler's No. 15.*

Carbonic-acid gas, 11.35 per cent.	
Bicarbonate of soda.....	22.51
Alum.....	20.38
Starch.....	29.38
Hydrate of alumina, sodium sulphate, ammonium sulphate, moisture, etc.....	27.73
	100.00

26. *Carlton.*

Carbonic-acid gas, 6.60 per cent.	
Bicarbonate of soda.....	13.44
Alum.....	11.85
Starch.....	43.77
Hydrate of alumina, sodium sulphate, ammonium sulphate, moisture, etc.....	30.94
	100.00

27. *Gem.*

Carbonic-acid gas, 8.45 per cent.	
Bicarbonate of soda.....	16.13
Alum.....	15.17
Starch.....	32.13
Hydrate of alumina, sodium sulphate, ammonium sulphate, moisture, etc.....	36.57
	100.00

28. *Scioto.*

Carbonic-acid gas, 8.80 per cent.	
Bicarbonate of soda.....	16.80
Alum.....	15.80
Starch.....	49.15
Hydrate of alumina, sodium sulphate, ammonium sulphate, moisture, etc.....	18.25
	100.00

29. *Zipp's Grape Crystal.*

Carbonic-acid gas, 10.90 per cent.	
Bicarbonate of soda.....	22.49
Alum.....	19.57
Starch.....	45.95
Hydrate of alumina, sodium sulphate, ammonium sulphate, moisture, etc.....	11.99
	100.00

30. *Forest City.*

Carbonic-acid gas, 7.80 per cent.	
Bicarbonate of soda.....	15.73
Alum.....	13.63
Starch.....	46.60
Hydrate of alumina, sodium sulphate, ammonium sulphate, moisture, etc.....	24.04
	100.00

Since the issuance of the foregoing circular the manufacturers of certain of these brands of baking-powders have sought to pervert the facts brought out by these analyses, and, by arguments and conclusions wholly unwarranted by the facts stated, or by the principles of chemical science, have for their own benefit held and assumed that the circular and analyses show a state of facts which they do not show, and lead to conclusions to which they do not lead. Nevertheless, the truth will assert itself, and this investigation and discussion, which is still going on, will throw a flood of light on this whole field of commercial food products that will be of incalculable benefit to the people of the State.

Professor Weber's analyses are rather superficial and incomplete, probably being made under conditions that did not admit of thorough quantitative work. He has overlooked entirely, for instance, the fact of the presence of phosphoric acid in many alum powders.

PROFESSOR CORNWALL'S REPORT ON BAKING-POWDERS.

Following are the results of an examination of a large number of baking-powders by Prof. H. B. Cornwall, together with his description of the methods of analysis employed, and his observations and conclusions.¹

METHODS OF ANALYSIS.

The analysis was directed toward determining the "strength" of the powders, or their yield of carbonic-acid gas, and their composition, so far as to indicate the nature of the chief active constituents. No great importance was attached to the amount of starch or other legitimate "filling," which only has an effect on the strength of the powder, nor was it possible to examine so large a number of samples minutely as to the residues left by them. Especial attention was therefore paid to the presence of possible objectionable constituents of the residues, and to ingredients that might render the use of the powders injurious.

Carbonic-acid gas.—This was determined with great care by boiling 1 gram (15.43 grains) of the powder with 125 to 130 cubic centimeters (about $4\frac{1}{2}$ fluid ounces) of distilled water in a roomy flask, connected with a Classen condensing, drying, and absorbing apparatus (Classen, Quantitative Chemische Analyse, 1885), the carbon dioxide being absorbed in soda-lime tubes, of which there were two, having their further ends charged with carefully-dried chloride of calcium. The contents of the flask were boiled, with proper use of a slow current of air, for one and one-half to one and three-quarters hours, and the current of air was kept up for half an hour after removing the flame, so that the whole operation lasted from two to two and one-half hours. Only in this way was the carbonic-acid gas with certainty to be expelled from the somewhat viscid, starchy water solution and completely carried over into the absorption tubes.

Tested before the analyses were begun, with a sample of probably one of the best commercial bicarbonates of soda in the market, the absorption apparatus yielded 51.38 per cent. of carbon dioxide; measurement of the gas (see below) indicating 51.44 per cent.

Tested in the course of the series of analyses by decomposing Iceland spar (crystallized carbonate of lime) with citric acid in the presence of starch, in the proportion used in the average of good cream of tartar baking-powders, the absorption apparatus showed 43.83 per cent., theory requiring, for absolutely pure carbonate of lime, 44 per cent.

As a check analysis, when it could be properly done, the gas evolved from the powder by 10 cubic centimeters of a mixture of one volume of hydrochloric acid, specific gravity 1.2, with two volumes of water, in a Scheibler's evolution bottle, was collected over mercury and measured, correction being made for atmospheric pressure, temperature, and moisture, and also an allowance for the carbon dioxide retained by acid of the strength used, as determined by tests with the Iceland spar. Enough baking-powder was taken to give 90 to 110 cubic centimeters of gas. The results by measurement averaged 0.12 per cent. below those by absorption and weighing of the gas, probably on account of the difficulty of liberating the gas, even by violent shaking, from the somewhat viscid liquid produced by the action of the strongly acid solution on the starch of flour. The greatest difference by the two methods was 0.29 per cent.

Whenever a sample showed a rather low percentage of carbonic-acid gas, or left a

¹ Report of the Dairy Commissioner of New Jersey, 1888, p. 82.

decidedly alkaline solution, duplicate tests were made by the soda-lime absorption, and no dependence was placed on measurement, but in other cases it was a most convenient and reliable check on the other method.

Sulphates.—These were detected in the cold-water solution of the baking-powder, bearing in mind the possible solvent action of citric acid on the barium sulphate. No attention was paid to minute quantities of soluble sulphates.

Ammonia salts.—These were detected by rubbing the powders with water and slaked lime, after ascertaining that ordinary samples of flour gave no reaction for ammonia under the conditions of our tests. No notice was taken of ammonia unless the turmeric paper was rapidly and decidedly colored.

Phosphates.—It was found that even in the presence of tartaric acid these could generally be detected by means of ammonium molybdate in the solution of the powder in very dilute nitric acid. In cases of doubt, the powder was first fused with carbonate of soda and nitrate of potash.

Alumina.—Although it could always be detected in the solution of the powder in very dilute nitric acid, at least, by the aid of acetic acid and phosphate of soda, yet all of the powders were also tested by fusion with carbonate of soda and nitrate of potash, extraction with boiling water, acidifying the filtered solution with hydrochloric acid and precipitating with ammonia water. The alumina in the precipitate was identified as such, however obtained. During the fusion abundant evidence of the presence of iron compounds as an impurity in the alum powders was frequently obtained, showing carelessness or ignorance on the part of the makers.

Tartaric acid and tartrates.—Free tartaric acid was dissolved out by absolute alcohol and identified. Tartrates were systematically tested for in case of doubt, but, in general, it was deemed sufficient to confirm their presence by shaking the powder with ammonia water, filtering, adding a crystal of nitrate of silver, and heating gently to form the characteristic silver mirror. It was found that phosphates and citrates did not interfere with this test when any considerable quantity of tartrates was also present in the solution, but it was depended on only as confirmation of the presence of tartrates in the cream of tartar powders.

Potash.—This was detected by holding some of the powder on a platinum wire in the Bunsen-burner flame and observing the flame coloration through a solution of permanganate of potash so strong as scarcely to transmit diffused daylight. Unless a decided red flame coloration was obtained, potash was certainly absent in any notable quantity.

RESULTS OF ANALYSIS.

The following tables give the results of the analysis of our samples, so far as was necessary to classify them and determine their "strength," that is, the percentage of carbonic-acid gas. The cubic inches of gas are given from 1 ounce avoirdupois of powder, at a temperature of 60° F., and barometer at 30 inches:

I. *Cream of tartar powders.*—In this class are placed all powders giving reactions for tartaric acid and potash, and free from phosphates, alumina, and any considerable quantity of soluble sulphates. Ammonia was sometimes present; whether as sesquicarbonate or bitartrate was not determined. Free tartaric acid was found in one case. Its presence has no effect on the wholesomeness of the powder, nor has the small amount of ammonia in any case found. The writer's experience is that the powders free from ammonia compounds yield just as light biscuits, etc., as the others.

As regards purity of materials, there seems little choice between the higher grades of these powders.

II. *Acid phosphate of lime powders.*—The first two of these were packed in tightly-corked glass bottles, and contained enough starchy material to keep them from deteriorating in these bottles.

The bread preparation consisted of two separate powders, each in a paper package. One was bicarbonate of soda, the other acid phosphate of lime mixed with starch. The strength was determined on a mixture of the two in the proportions directed on the packages.

The wheat powder was put up in tin boxes, without starch or other filling. One sample was in excellent order, the other much caked.

III. *Alum and phosphate powders*.—This class embraces powders showing ammonia, soluble sulphates, alumina, and phosphates, when tested as already described.

A few showed potash reactions, and in some there was evidence of tartaric acid or some other substance reducing silver abundantly from ammoniacal solutions. In such cases, of course, potash alum and bitartrate of ammonium may have been present, or the reactions may have been caused by cream of tartar, or by free tartaric acid. The possible combinations are very numerous, and the analysis, however complete, will not always indicate the exact combination. Inasmuch as some of these powders showed considerable alumina in the simple water solution, a more detailed examination of them is recommended, for the reasons already given. The actual presence of acid phosphate of lime, or of any other acid phosphate, was not proven, but all contained some phosphate, and have therefore been classed as indicated, although probably in every case they were made with acid phosphate of lime.

As already mentioned, the low grade of several is, perhaps, from deterioration, due to the presence of the acid phosphate in packages not sufficiently air-tight. Acid phosphate will not keep well when mixed with bicarbonate of soda, except in well-corked bottles. Tin cases are not tight enough.

Many of these powders contained sulphate of lime, chemically equivalent to *terra alba*. This was, perhaps, in no case added as an adulterant, but was a part of the acid phosphate of lime used; the latter not having been separated from the sulphate of lime formed in its manufacture. The presence of this sulphate of lime must be regarded as objectionable. None of these powders are as strong as they might be made, and most of them are very deficient in strength. Apart from questions of healthfulness, there can be no economy in buying some of these powders.

IV. *Alum powders*.—Here are classed the powders showing the same reactions as the preceding class, but free from phosphates. All appeared to be ammonia alum powders, but reactions for potash and tartaric acid were not wanting among them. Only one of them begins to come up to the strength which a "straight" burnt ammonia alum powder might have.

V. *Unclassed powders*.—The composition of these will be indicated under the special remarks.

I.—Cream of tartar powders.

No.	Brand.	Carbonic-acid gas.	Cubic inches carbonic-acid gas per ounce.	Remarks.
		<i>Per cent.</i>		
4	The Best	11.60	107.3	
4	Sea Foam	10.86	100.5	Yields a little ammonia and soluble sulphate.
23	Sterling	11.70	108.2	Yields ammonia reactions.
29	Health	6.96	64.44	Final reaction of aqueous solution strongly alkaline. See special remarks.
50	Health	7.25	67.1	
39	None Such	12.64	116.9	
40	Cleveland's	13.27	122.7	Received in June.
43	Cleveland's	13.82	127.8	Received in November.
41	Royal	13.56	125.43	Yields ammonia reactions. Received in May.
42	Royal	13.06	120.8	Yields ammonia reactions. Received in November.
45	Price's "Cream"	11.95	110.5	Received in May. Contains free tartaric acid.
53	Price's "Cream"	12.20	112.9	Received in December.
	Average, 8 brands	11.60	Excluding 29 and 50, average is 12.46 per cent. of carbonic acid.

II.—*Acid phosphate of lime powders.*

No.	Brand.	Carbonic-acid gas.	Cubic inches carbonic-acid gas per ounce.	Remarks.
		<i>Per cent.</i>		
46	Horsford's Phosphatic	14.95	138.3	Received in August. In 8-ounce glass bottle.
54	Horsford's Phosphatic	14.01	129.6	In retail dealer's stock one year. A little gas escaped on opening the 4-ounce bottle.
47	Rumford's Yeast Powder	13.51	125.0	Received in May. In 8-ounce glass bottle.
48	Rumford's Yeast Powder	13.89	128.5	Received in August. In 8-ounce glass bottle.
49	Horsford's Bread Preparation	15.39	142.4	Received in August. Bicarbonate soda and acid phosphate put up in separate papers. The acid phosphate was not quite free from soluble sulphates.
21	Wheat	15.62	144.5	In tin box; in good order.
52	Wheat	5.83	53.9	In tin box; much caked.

NOTE.—Since the rapidity with which a baking-powder gives off carbonic-acid gas in the cold varies with the ingredients used, it was deemed worth while to test some powders as follows: Forty-five grains (3 grams) of each was mixed with as little shaking as possible with $\frac{1}{8}$ ounce (5 cubic centimeters) of water, and the volume of gas evolved in five minutes was measured.

	Per cent.
Cleveland's yielded of its carbonic acid.....	49.6
Royal yielded of its carbonic acid.....	45.6
Horsford's yielded of its carbonic acid.....	68.8
A "straight" burnt alum powder yielded of its carbonic acid.....	6.3

III.—*Alum and phosphate powders.*

No.	Brand.	Carbonic-acid gas.	Cubic inches carbonic-acid gas per ounce.	Remarks. (All give ammonia reactions.)
		<i>Per cent.</i>		
1	Patapsco	8.32	77.0	
2	Washington	8.81	82.5	Received in May.
27	Washington	9.97	92.2	Received in November.
3	Davis's O. K.	8.99	83.2	
7	McDowell's G. and J.	9.70	89.7	
9	Lincoln	9.73	90.0	
10	Kenton	7.01	64.8	Received in October. Another sample received in May gave 3.81 per cent.
11	State	6.70	62.0	Received in October.
15	State	8.42	77.9	Received in May.
13	On Top	9.17	84.8	
16	Perfection	5.09	47.1	In pasteboard box with tin ends.
19	Silver Star	9.51	88.0	
24	Our Own	10.47	96.8	
35	White Star	10.09	93.3	
28	Somerville	8.39	77.6	
30	Grape	10.02	92.7	
31	Sovereign	8.96	82.9	
32	A. and P. (Atlantic and Pacific)	8.97	83.0	
33	Higgins	6.63	61.3	Received in September.
51	Higgins	11.30	104.5	Received in December.
34	Windsor	8.77	81.1	
37	Brooks and McGeorge	10.16	94.0	
38	Henkel's	10.24	94.7	
Average, 20 brands		8.97		

IV.—*Alum powders.*

No.	Brand.	Carbonic-acid gas.	Cubic inches carbonic-acid gas per ounce.	Remarks. (All show ammonia reactions.)
		<i>Per cent.</i>		
8	Miles's "Prize"	9.63	89.1	Shows potash reactions and reduces silver abundantly.
20	Four Ace	10.31	95.4	
26	Feather Weight	9.63	89.1	Two other samples gave respectively 15.35 and 16.73 per cent.
36	One Spoon	16.77	155.1	

V.—*Unclassed powders.*

No.	Brand.	Carbonic-acid gas.	Cubic inches carbonic-acid gas per ounce.	Remarks.
		<i>Per cent.</i>		
6	Silver Prize	8.14	75.3	Shows potash and ammonia reactions, and reduces silver abundantly.
22	Orange	8.00	74.0	
18	Our Best	6.15	56.9	Shows ammonia reactions; contains much soluble sulphates, and some free tartaric acid.

SPECIAL REMARKS.

Sample No. 6.—This sample shows strong reactions for ammonia, soluble sulphates, soda, and potash. Its aqueous solution, rendered ammoniacal before filtering, reduces silver from a crystal of the nitrate, as a bright coating on the glass. It would have been classed among the cream of tartar powders had it not shown altogether too much soluble sulphates. Shaken with absolute alcohol it renders this slightly alkaline; boiled with water the powder gives a decidedly alkaline solution. It contains some alumina.

Sample No. 18.—This gave strong reactions for ammonia, soluble sulphates, soda, and potash. Tartaric acid was extracted from it by shaking with absolute alcohol. It may contain some cream of tartar, but has too much soluble sulphates to warrant placing it among the cream of tartar powders.

Sample No. 22.—This powder gave strong reactions for alumina, soluble sulphates, and soda. Neither potash nor ammonia was present. The label stated that it contained grape (tartaric?) and orange (citric?) acids, combined "with natron, bicarb. soda, and corn starch," and the analysis indicated the presence of tartrates and citrates, as well as much alumina, which was abundantly found in the aqueous solution of the powder even after boiling with the undissolved residue. Alumina in a soluble form was also extracted in considerable quantity by water alone from bread made with this powder. Apparently, the organic acids kept it in soluble condition. Since neither potash nor ammonia was present, the alumina appears to have been added in the shape of sulphate of alumina, or else of soda alum.

After washing away the starchy matter with chloroform and examining the residue under the microscope in polarized light, crystalline fragments of a singly refracting substance were observed in abundance, together with doubly refracting crystalline

materials. Although no octahedral crystals could be distinctly made out, yet there were some fragments of soda alum, which is as good as any other alum for making baking-powders, so far as chemical and physiological effects are concerned. It is more likely to be affected by moisture than "burnt alum."

Sample No. 29.—This powder was very strongly alkaline, containing so great an excess of bicarbonate (or carbonate) of soda that if the proper amount of cream of tartar had been used the powder would have yielded about 11 per cent. of carbonic-acid gas.

CONCLUSIONS.

Our investigations show that while especially the higher grades of cream of tartar and acid phosphate of lime powders are maintained at a quite uniform standard of excellence, the State is flooded, also, with many baking-powders of very poor quality—cheap goods, poorly made. Of the thirty-nine brands examined, twenty-five contain alum or its equivalent, in the shape of some soluble alumina compound; eight are cream of tartar powders, with small quantities of other ingredients in several cases; four are acid phosphate of lime powders; two belong properly under none of the above classes.

With one exception, the powders containing alum all fall below the average strength of the cream of tartar powders, and in the majority of cases they fall much below the better grades of the cream of tartar powders.

In the cream of tartar and the acid phosphate of lime powders, no indications of substances likely to be injurious to health, in the quantities used, have been found.

More evidence against the use of alum in baking-powders might have been presented, but it would have been of a similar nature to that which has already been given. In the writer's opinion, the presence of alum in baking-powders is objectionable, since, under certain conditions, it may exert an injurious effect on the digestion. The effects may not be very marked in the case of any individual consumer, but that they can be induced to a greater or less extent seems to be well established.

There appears to be ample ground for requiring that the makers of baking-powders should publish the ingredients used in their powders, in order that the consumer, who may justly have doubts of the desirability of using certain kinds, may be protected. At present the only guaranty of an undoubtedly wholesome and efficient article appears to be the name of the brand.

Moreover, since it is quite possible to put up the baking-powders in such a way as to preserve their strength very thoroughly, and since it is evident that many makers fail in this respect, it would not seem unreasonable to require that baking-powders should not be sold unless they will yield a certain percentage of carbonic-acid gas. The bad effects of the "heavy" food prepared with some of the baking-powders among our samples must certainly be felt by those who use them, and who are yet too ignorant to know where the trouble lies. It is for this class especially that nearly all legislation relating to securing good food and drugs is enacted.

Since it is evident that some of the alum powders are so prepared as to increase the extent of any injurious effect, owing to the mixture of ingredients whose combination can not be justified on any grounds, it is recommended that a special and more thorough examination of such be made, with a view to preventing their manufacture.

APPENDIX.

1. In bread made from Orange baking-powder, page —, according to directions, there was found, in a condition readily soluble in tepid water, alumina equivalent to 26 grains of crystallized ammonia alum per 2-pound loaf.

2. With reference to Professor Patrick's experiments on cats, the writer had biscuit made with a "straight" alum baking-powder, using twelve times the proper amount of the powder. The biscuit had a bitter, nauseating taste, and must have been very indigestible, so that no fair conclusions could be drawn from its use.

ANALYSES BY U. S. DEPARTMENT OF AGRICULTURE.

The samples for the following analyses were all purchased at retail stores in Washington, D. C., by an agent of the Department, no intimation being given to the seller of the purpose for which they were intended. The city was pretty thoroughly gone over, and the samples probably include about all the different brands sold.

METHODS OF ANALYSIS.

Following are detailed descriptions of the methods followed in the analyses made in this laboratory. In many of the estimations different methods were tried, and the one which gave the best results and was found to be most convenient was chosen.

GENERAL EXAMINATION OF SAMPLES.

The qualitative and general examination is described in the extract from Professor Cornwall's report above, and it is not necessary to go into it in detail again, as the methods were similar, generally speaking. The qualitative examination and assignment of a sample to one of the classes indicated presents no special difficulties. If it is desired to know the character of the filling used, it is readily ascertained by a microscopical examination; but this is rather an unimportant matter. A determination of the alkalimetry of the watery solution of the powder is useful as showing whether any great excess of alkali had been used.

ESTIMATION OF CARBONIC ACID.

This is one of the most important estimations, as it determines the strength of the powder. It was made by absorption with soda lime, and a form of apparatus was used that has served for some time in this laboratory for the determination of carbonic acid. This apparatus has recently been somewhat modified and greatly improved in compactness by Mr. A. E. Knorr. It is shown in the accompanying figure. Following is Mr. Knorr's description:¹

The apparatus proper, as represented by the cut, consists of a flask A in which the carbon dioxide is set free. A condenser D is ground into the neck of this flask and condenses the steam formed when the liquid in A is boiled in order to secure complete expulsion of the gas. The reservoir B contains the acid required for the operation, and has a soda-lime guard C ground into it to retain the carbonic acid of the air, a constant current of which is aspirated through G during the whole operation. The stem of the reservoir is ground into the condenser, or it may be conveniently blown into one piece with it. The carbonic acid is dried in E and finally absorbed in the weighed potash bulb F.

Two determinations of carbonic acid were made on each sample—one by the addition of acid to determine the total amount of carbonate pres-

¹ Jour. Analyt. Chem., 3, 162.

ent, and one by the addition of water only. The per cent. of carbonic acid found in the latter estimation may properly be termed the *available* amount present in the powder, as it is the quantity which would be actually liberated by the acid ingredient of the powder when it is used in baking, and therefore represents the actual *value* of the powder for aerating purposes, so far as the *evolution* of gas is concerned.

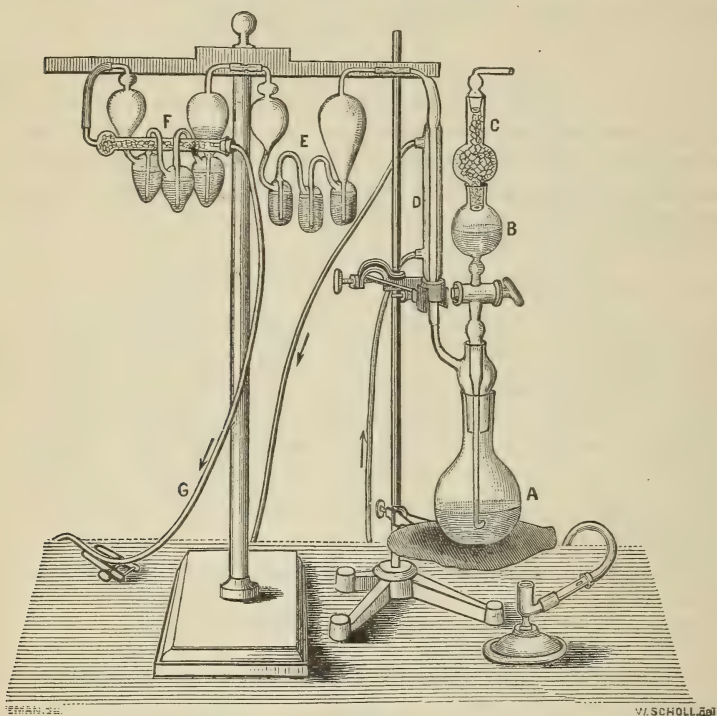


FIG. 26.

For the determination of the *total* CO_2 the procedure was as follows: Place in a short glass tube, the weight of which is known, about 1 to 2 grams of the sample, and weigh the whole as quickly as possible, the amount taken being obtained by difference. The tube and contents are gently dropped into the generating flask of the apparatus, which must be perfectly dry. The flask is closed with the stopper carrying the tube connecting with the absorption apparatus, and also the funnel tube, which has been previously provided with 10 cubic centimeters of dilute sulphuric acid for the liberation of the gas. When all parts of the apparatus are connected, and seen to be tight, the stopper of the funnel tube is opened, and the acid allowed to run slowly into the flask. The generation of the gas should be made as gradual as possible; by running in a small quantity of the acid at first and tilting the flask slightly this can readily be accomplished; after the greater part of the sample has been acted upon by the acid and before the latter has all been added,

a lamp is placed under it, and the contents gradually heated to boiling, gentle aspiration being made at the same time. The operation is finally finished by the funnel tube being opened, and air, free from CO_2 , drawn through it and through the apparatus, the contents of the flask at the same time being kept at ebullition. This is continued for fifteen minutes, when the absorption tube is removed from the apparatus, allowed to cool, and weighed. Its increase in weight gives the amount of CO_2 absorbed. The determination of the *available* CO_2 was conducted in a similar manner, with the substitution of pure boiled water in the funnel tube instead of acid. After the sample has all been acted on, the contents of the flask are just brought to a boil, then the lamp is removed and air is drawn through the liquid for exactly fifteen minutes. The conditions were made as nearly alike as possible for each sample in this estimation, for, different results can readily be obtained by varying them. The above conditions are believed to be as close an approximation to those actually obtaining in the use of the powder as can be arrived at in an ordinary chemical analysis. Prolonged boiling of the liquid residue is inadvisable, for in case the ingredients in the powder are not accurately proportioned, and a considerable excess of bicarbonate is present, long boiling will liberate gas from it after the acid ingredient has all been neutralized, and thus a high result is obtained from a poorly-made sample, while in one where the bicarbonate is not greatly in excess of the proper amount, the above procedure will readily give the full amount available. *

*In some experiments made to determine the amount of carbonic acid driven off from bicarbonate of soda on heating its water solution, the following results were obtained: (1) Just brought to a boil under the same conditions as in the determinations made above, 6.99 per cent. of the weight of the bicarbonate was obtained; (2) Boiled 15 minutes, 16.17 per cent. was obtained; and, (3) boiled $1\frac{1}{2}$ hours, 20.70 per cent., or about the full quantity of acid carbonate.

ESTIMATION OF STARCH.

This estimation was made by the well-known method of conversion by heating with acid and the determination of the copper oxidereducing power of the resultant solution. While by no means satisfactory, this is probably the best method we have at present for starch estimation. No difficulty was found in its application to all classes of baking-powders, the other ingredients offering no obstacle to its proper performance. To insure agreeing results it is very essential to conduct the conversion under precisely the same conditions in all cases.

The following is the detailed procedure:

From 2 to 5 grams are weighed out and transferred to an Erleumeyer flask; to it are added about 150 to 200 cubic centimeters of a solution of hydrochloric acid which has a strength of 4 per cent. of the acid gas. The flask is provided with a cork, perforated for the passage of a condensing tube about 1 meter in length. The conversion is accomplished

by gently boiling the acid liquid for four hours, after which the flask and contents are cooled, neutralized by the addition of sodium hydrate, made up to a definite volume and the copper oxide reducing power determined. The latter operation is best carried out by the method used in this laboratory, in which asbestos-tipped filtering tubes are used for the end reaction.¹ The reducing power being calculated as dextrose; 10 parts equal 9 parts of anhydrous starch.

Professor Weber used a rough method for the direct estimation of starch in his samples, which he describes as follows:²

One gram was weighed off, transferred to a small beaker, covered with water, allowed to stand until action had ceased, filtered and washed, residue spurted by means of a wash-bottle into a flat-bottomed platinum dish, allowed to settle, the supernatant water removed as far as possible by means of a pipette, the remainder of water evaporated, the residue dried at 100° C. and weighed. The residue was then incinerated and the amount of ash deducted from above weight. In case of alum powders the ash remaining after ignition was Al_2O_3 , which was contained in the residue dried at 100° C. as $\text{Al}_2(\text{OH})_6$; consequently the Al_2O_3 was calculated as $\text{Al}_2(\text{OH})_6$ before deducting.

This method was carried out upon the entire series of samples examined here. In many cases it gave results agreeing quite closely with those obtained by the direct estimation, but in some samples the results were entirely too high. It is not applicable to alum powders even with the correction made above. For a rough method it answers fairly well and it is quite easy of execution.

ESTIMATION OF PHOSPHORIC ACID.

This determination was made in the same manner as in fertilizers, as prescribed by the Association of Official Agricultural Chemists at their last meeting, as follows:³

Weigh out 2 grams of the sample, ignite carefully in a muffle, and treat with 30 cubic centimeters concentrated nitric acid.

Boil gently until all phosphates are dissolved and all organic matter destroyed; dilute to 200 cubic centimeters; mix and pass through a dry filter; take 50 cubic centimeters of filtrate; neutralize with ammonia. To the hot solutions for every decigram of P_2O_5 that is present add 50 cubic centimeters of molybdic solution. Digest at about 65° C. for one hour, filter, and wash with water or ammonium nitrate solution. (Test the filtrate by renewed digestion and addition of more molybdic solution.) Dissolve the precipitate on the filter with ammonia and hot water and wash into a beaker to a bulk of not more than 100 cubic centimeters. Nearly neutralize with hydrochloric acid, cool, and add magnesia mixture from a burette; add slowly (one drop per second), stirring vigorously. After fifteen minutes add 30 cubic centimeters of ammonia solution of density 0.95. Let stand several hours (two hours

¹ Fully described in Bull. No. 15, p. 32.

² Communicated to the author in MSS.

³ Bull. No. 19, Chem. Div. U. S. Dep't Ag'l, p. 58.

are usually enough). Filter, wash with dilute ammonia, ignite intensely for ten minutes, and weigh.

ESTIMATION OF TARTARIC ACID.

The method used in this estimation was that known as the "Goldenberg Geromont," which is described in full in *Chemiker Zeitung*, 12, 1888, 390. This and other methods for the estimation of tartaric acid in crude argol and other raw materials were lately submitted to a critical comparison in the tartrate factory at Nienburg;¹ the Goldenberg-Geromont method avoided the principal sources of error and is recommended as the best and most easy of execution.

The procedure as modified for application to a tartrate baking-powder is as follows:

Weigh out 5 grams, wash into a 500-cubic centimeter flask with about 100 cubic centimeters of water; add 15 cubic centimeters of strong hydrochloric acid; make up to mark and allow the starch to settle. Filter, measure out 50 cubic centimeters of the clear filtrate; add to it 10 cubic centimeters of a solution of carbonate of potash containing 300 grams K_2CO_3 to the liter and boil half an hour; filter into a porcelain dish and evaporate filtrate and washings to a bulk of about 10 cubic centimeters. Add gradually with constant stirring 4 cubic centimeters glacial acetic acid, and then 100 cubic centimeters of 95 per cent. alcohol, stirring the liquid until the precipitate floating in it assumes a crystalline appearance. After it has stood long enough for this precipitate to form and settle, best for several hours, decant through a small filter, add alcohol to the precipitate, bring it on the filter, wash out the dish and finally the filter carefully, with alcohol, until it is entirely free from acetic acid. Transfer filter and precipitate to a beaker, add water and boil, washing out the dish also with boiling water if any of the precipitate adheres to it. The resulting solution is titrated with decinormal alkali solution, using phenol-phthalein as indicator; 1 cubic centimeter decinormal alkali corresponds to .0188 grams of potassium bitartrate, or .0150 grams of tartaric acid.

ESTIMATION OF ALKALIES.

The estimation of the soda and potash in the powders was carried out by separating them as chlorides, determining the potash as potassium platonic chloride and calculating the difference as sodium chloride. The detailed procedure was similar to that used by the Association of Official Agricultural Chemists for determining potash in fertilizers, as follows:

Weigh out 5 grams into a platinum dish and incinerate in a muffle at a low heat. The charred mass is well rubbed up in a mortar, then boiled 15 minutes with about 200 cubic centimeters of water to which

¹ *Chemiker Zeitung* 13, 1889, 160.

has been added a little hydrochloric acid. The whole is transferred to a 500-cubic centimeter flask and after cooling made up to the mark and filtered. Of the filtered liquid 100 cubic centimeters, representing 1 gram of the sample, are measured out, heated in the water bath, and slight excess of barium chloride added; then without filtering barium hydrate is added in slight excess, the precipitate filtered off and washed. To the filtrate is added a little ammonium hydrate and then ammonium carbonate until all the barium is precipitated. This precipitate is filtered and washed, the filtrate evaporated to dryness and carefully ignited until all volatile matter is driven off, when it is weighed. This gives the weight of the mixed chlorides. The residue is taken up with hot water, from 5 to 10 cubic centimeters of a 10 per cent. solution of platinic chloride added, and the whole evaporated to a sirupy consistence in the water bath; then it is treated with strong alcohol, the precipitate washed with alcohol by decantation, transferred to a Gooch crucible, dried at 100° C., and weighed. The weight of the precipitate multiplied by .19308 gives the weight of K_2O , and by .3056 the equivalent amount of KCl . The weight of KCl found is subtracted from the weight of the mixed chloride, the remainder being the $NaCl$, which multiplied by .5300 gives the weight of Na_2O in the sample.

ESTIMATION OF ALUMINIUM.

In the case of a "straight" alum powder, the simple estimation by burning to ash, extracting, and determining the alum by direct precipitation with ammonia would probably be accurate, but in view of the frequent use of flour as a "filler," as well as of the presence of calcium as an impurity, it is best, even with those made up with alum alone, to use a method which will insure a complete separation of the alum. The following procedure, given by Allen for the quantitative estimation of alum in bread, was found to give good results with baking-powders:

Take 5 grams and incinerate in a muffle. The heat should be moderate so as not to fuse the ash. The process is completed by adding pure sodium carbonate and a little niter, and heating the mixture to fusion. The product is rinsed out with water into a beaker, acidulated with hydrochloric acid, and evaporated to dryness. The residue is taken up with dilute acid, the liquid made up to 500 cubic centimeters in a graduated flask, filtered, and 50 cubic centimeters taken for precipitation. To the solution dilute ammonia is added until the precipitate barely redissolves on stirring, when a slightly acid solution of ammonium acetate is added, and the liquid brought to a boil. After a few minutes' heating the solution should be set aside for some hours, when its appearance should be observed. (If gelatinous it probably consists solely of iron and alum phosphates, but if granular more or less of the earthy phosphates have been co-precipitated; then it should be separated, redissolved in dilute hydrochloric acid, the solution again neutralized with ammonia, and treated with ammonium acetate.) The pre-

precipitate of iron and aluminium phosphates is filtered off, washed, and redissolved in the smallest quantity of hydrochloric acid. The resultant solution is poured into an excess of an aqueous solution of *pure* caustic soda contained in a platinum or nickel vessel. After heating for some time, the liquid is considerably diluted and filtered. The filtrate is acidulated with hydrochloric acid, ammonium acetate and a few drops of sodium phosphate added, and then a slight excess of ammonia. The liquid is kept hot till all smell of ammonia is lost, when it is filtered, and the precipitated aluminium phosphate washed, ignited, and weighed. Its weight multiplied by 3.713 gives the ammonia alum (hydrated), or by 3.873 the potash alum in the .5 grams of sample taken.

In the phosphate and alum powders the above method gave a fairly good separation of the alum, but the following separation by means of molybdenum was found to be more exact, and at the same time much more convenient of application. It was adapted to the powders by Mr. K. P. McElroy.

Weigh out 5 grams into a platinum dish, char, treat with strong nitric acid, and filter into a 500 cubic centimeter flask. After washing the residue slightly, transfer filter and all back to the platinum dish and burn to whiteness. To the ash add mixed carbonates and fuse. Take up with nitric acid, evaporate to dryness, acidify again with nitric acid, and wash all into the 500 cubic centimeter flask. Nearly neutralize the contents of the flask with ammonia, and add molybdate of ammonium sufficient to precipitate all the phosphoric acid present. Allow to stand some time, make up to the mark, shake thoroughly, and filter off 100 cubic centimeters through a dry filter. This is exactly neutralized with ammonia, keeping the solution as cool as possible to avoid the deposition of molybdic acid. Filter and wash the precipitate, redissolve in dilute nitric acid with the addition of a little ammonium nitrate, and precipitate as before. Filter through a paper filter, burn, ignite, and weigh as Al_2O_3 . The alumina and phosphoric acid may be determined in the same sample by the above method, modifying it as follows: When the solution, ash, etc., have all been brought into the graduated flask, make up to the mark without adding molybdate, filter and take 100 cubic centimeters, nearly neutralize with ammonia, add ammonium nitrate and molybdate of ammonium, digest and filter. The filtrate contains the aluminium and may be precipitated with ammonia as above, while the phosphoric acid is all contained in the precipitate, which may be redissolved in ammonia and precipitated with magnesia mixture.

ESTIMATION OF CALCIUM.

This determination was made as follows: Weigh out 5 grams of the sample, transfer to a 500 cubic centimeter flask, add 40 or 50 cubic centimeters of water, and then 20 or 30 cubic centimeters of strong hydrochloric acid. Make up to the mark with water, shake thoroughly, and set aside to allow starch to settle. Filter through a dry filter, and take

aliquot parts of the filtrate for precipitation ; in phosphate powders not more than 50 cubic centimeters should be used. Nearly neutralize with ammonia, acidify slightly with acetic acid, add ammonia acetate, and boil. Filter from the precipitate, if there be any, add ammonium oxalate, and allow to stand several hours. Filter into a Gooch crucible, and dry at 100°. Weigh as oxalate.

ESTIMATION OF SULPHURIC ACID.

The sulphuric acid was estimated without previous ignition of the powder, as follows :

Weigh out .5 to 1 gram of the powder, according to its character, the former quantity being more convenient for alum powders, and transfer to a beaker. Digest with strong hydrochloric acid until all of the powder, including the starch, goes into solution ; add barium chloride to slight excess while still hot, and allow it to stand for twelve hours, or over night. Filter through a Gooch crucible, ignite, and weigh.

ESTIMATION OF AMMONIA.

Ammonia is present either as bicarbonate, or as ammonium sulphate in the alum powders. The estimation was made by the Kjeldahl method, as used by the Association of Official Agricultural Chemists.¹ Where flour instead of starch is used as a filling the gluten would give ammonia, of course, and wherever a tartrate powder was found to give any appreciable amount of ammonia by the method, a weighed portion was taken, water added, the solution filtered from the starch, and subjected to analysis. The results were practically the same as those obtained directly from the powder. Probably flour is not often used. In the case of the alum powders, the difference that would be made by flour filling was disregarded, as the amount of alum present is sufficiently established by the percentage of aluminium oxide and sulphuric acid found ; the amount of ammonia found was almost invariably low in proportion to these other constituents of ammonia alum.

ESTIMATION OF MOISTURE.

The percentage of water of association and combination as given in the analyses was obtained by difference. A number of attempts were made to estimate it directly in the following way : A weighed portion of the sample was placed in a U tube, which was kept immersed in boiling water. At one end this tube was connected with a series of sulphuric acid wash-bottles, and at the other with weighed potash bulbs filled with sulphuric acid, and beyond these with an aspirator. In this way a current of dried air was drawn through the sample while it was kept at 100° C., and the water drawn from it in this way was absorbed by the sulphuric acid in the potash bulb, while the carbonic acid was drawn

¹ Fully described in Bull. No. 19, Chem. Div. U. S. Dep't Agriculture.

into the aspirator. The increase in weight of the potash bulbs gave the weight of water absorbed. It was found, however, that the sample would cake into a hard mass, through which a channel would form which would permit the passage of the current of dry air, without affecting the greater mass of the powder, and no exact results could be obtained. Some improvement was made by mixing the powder with dry oxide of zinc, so as to prevent the formation of a channel, but still the results were not at all satisfactory, and the attempt to make a direct estimation was finally abandoned. Even if the determination could be made exact, it is doubtful if all the water of combination could be obtained at 100°C. , especially in phosphate and alum powders, and probably a temperature high enough to accomplish this would effect a decomposition of the starch.

EXPRESSION OF THE RESULTS OF ANALYSIS.

The results of analysis are given, first, as acid and basic radicals in percentage composition while in the second part of each table an attempt has been made to combine these into salts showing the constitution of the powder. The difficulties attending this calculation of the probable combination of the acids and bases were so great that I was frequently tempted to give it up entirely and state only percentage composition. I finally concluded to insert the calculation with the proviso that it should be considered at best merely an approximation to the exact composition of the powder. The obstacles in the way of an exact calculation may be stated as follows: In the first place the amount of total carbonic acid found is always less than that required to form bicarbonate of soda with the amount of sodium oxide found. This is undoubtedly due to a partial action of the acid constituent of the powder upon the bicarbonate, by which carbonic acid has been lost. The percentage of bicarbonate is therefore calculated from the per cent. of carbonic acid found, and the excess of sodium oxide left is stated as "residual" sodium oxide, without attempting to make further hypotheses as to the results of its combination with the acid constituent. It is possible that part of the bicarbonate may become converted into the normal carbonate under the conditions of being mixed and in contact with other chemicals, though this is not likely; then the indefinite composition of many of the commercial salts used in the powders renders it an extremely difficult matter to arrive at any satisfactory conclusion as to the make up of the powder in which they are used. The acid phosphate of lime, for instance, is a very variable substance, and even ammonia alum, which might reasonably be supposed to be constant in its composition, is found to vary widely from the theoretical. Its content of water varies according to the greater or less amount of drying it has undergone, and aside from this the ratios of the ammonia, sulphuric acid, and alumina to one another are at variance with the formula.

This is shown by the following analysis of a sample of commercial ammonia alum obtained in a powdered condition :

Analysis of commercial ammonia alum.

Constituents.	Found.	Theoretical composition.
	<i>Per cent.</i>	<i>Per cent</i>
Aluminium oxide, Al_2O_3	12.62	11.36
Sulphuric acid, SO_3	34.17	35.28
Ammonia, NH_3	2.75	3.75
Water of crystallization (by difference).....	50.46	49.61
	100.00	100.00

This indicates that the commercial salt is somewhat basic as regards the alumina, yet there is a deficiency of ammonia, so that if the former is all combined with sulphuric acid as normal sulphate, there is still not sufficient of the acid left for combination with the ammonia, although the latter is present in too low a proportion to the other constituents. This anomaly holds good throughout many of the samples.

I have expressed the sum total of the alumina, sulphuric acid, and ammonia as "anhydrous ammonia alum," combining the sulphuric acid, first with the alumina as far as it went, and the rest with the ammonia, and where there was not sufficient for combination with all the ammonia adding the latter *as ammonia*.

The presence of acid phosphate of lime still further complicates this calculation, as it is a question how much of the sulphuric acid should be taken from the alum to combine with part of the lime as sulphate of lime.

In the expression of the results where acid phosphate of lime is present, I have combined the lime with phosphoric acid as normal phosphate as far as it went, and added the rest as free phosphoric acid, grouping the whole together and calling it "acid phosphate of lime." Following is an analysis of a sample of commercial acid phosphate of lime, obtained from the trade :

Analysis of commercial acid phosphate of lime.

	<i>Per cent.</i>
Calcium oxide, CaO	24.93
Phosphoric acid, P_2O_5	52.45
Sulphuric acid, SO_315
Water	22.80
	100.33

In this sample the ratio of lime to phosphoric acid is about 1 : 2, and this relation holds good in many of the powders containing the phosphate, but in some it is quite different. The above sample is almost free from sulphate of lime, while many of the powders show considerable quantities of it, indicating that all the acid phosphates are

not so pure in this respect. Chemists will readily understand the impossibility of giving the proportions of the various forms of lime phosphates contained in such a substance. As given in the tables the relative acidity is shown, though of course the phosphoric acid does not occur as free acid.

Both the alum and the lime phosphate contain large percentages of water, hygroscopic moisture in the latter, and crystallization water in the former, so that the per cents. of the "anhydrous" salts given are always lower than the proportions of the hydrated salts originally used in compounding the powder. Nearly half the weight of the alum is crystallization water, some part of which is probably driven off in some cases when it is used for baking-powder purposes, but there are no means of ascertaining how much, and of course the moisture in the acid phosphate would vary in different samples, so there is no possible way of approximating the amounts of the hydrated substances, as they were originally used.

Ammonia bicarbonate is another substance of indefinite composition. As given in the tables it has been calculated from the ammonia found, upon the assumed composition given it in the U. S. Pharmacopœia, viz, $\text{NH}_4\text{HCO}_3 \cdot \text{NH}_4\text{NH}_2\text{CO}_2$.

In case ammonia carbonate were present in any of the powders containing ammonia alum, I know of no way of estimating the amount or even the fact of its presence in the small quantities used.

The percentage of "available carbonic acid" is placed first in the tables as constituting the most important indication of the efficiency of the sample as an aerating agent.

TARTRATE POWDERS.

5503.—*Roya Baking-Powder.*

[Manufactured by The Royal Baking-Powder Company, New York]

Available carbonic acid	per cent..	12.74
Cubic inches per ounce of powder at 212° F.....		153.0
Leavening gas (available $\text{CO}_2 + \text{NH}_3$)	per cent..	13.06
Cubic inches per ounce of powder at 212° F.....		160.6

PERCENTAGE COMPOSITION.

Total carbonic acid, CO_2	12.92
Sodium oxide, Na_2O	10.30
Potassium oxide, K_2O	12.02
Calcium oxide, CaO13
Ammonia, NH_332
Tartaric acid, $\text{C}_4\text{H}_4\text{O}_5$	37.46
Sulphuric acid, SO_325
Starch	16.34
Water of combination and association by difference	10.26
	<hr/> 100.00 <hr/>

PROBABLE PERCENTAGE COMBINATION.

Sodium bicarbonate, NaHCO_3	23.61
(Residual sodium oxide, Na_2O)	1.59
Ammonium bicarbonate, $\text{N}_3\text{H}_{11}\text{C}_2\text{O}_5$98
Potassium bitartrate, $\text{KHC}_4\text{H}_4\text{O}_6$	53.34
Calcium sulphate, CaSO_431
Starch	16.34
Water of association	3.83
	<hr/> 100.00

This powder contains a small quantity of ammonium bicarbonate.

5504.—*Dr. Price's Cream Baking Powder.*

[Made by Price Baking-Powder Company, New York and Chicago.]

Available carbonic acid	per cent..	11.13
Cubic inches per ounce of powder at 212°F		133.6

PERCENTAGE COMPOSITION.

Total carbonic acid, CO_2	12.25
Sodium oxide, Na_2O	11.03
Potassium oxide, K_2O	11.71
Calcium oxide, CaO19
Tartaric acid, $\text{C}_4\text{H}_4\text{O}_5$	35.14
Sulphuric acid, SO_312
Starch	18.43
Water of combination and association by difference	11.13
	<hr/> 100.00

PROBABLE PERCENTAGE COMBINATION.

Sodium bicarbonate, NaHCO_3	23.38
(Residual sodium oxide, Na_2O)	2.40
Potassium bitartrate, $\text{KHC}_4\text{H}_4\text{O}_6$	50.04
Calcium sulphate, CaSO_420
Starch	18.43
Water of association	5.55
	<hr/> 100.00

5505.—*Cleveland's Superior Baking-Powder.*

[Made by Cleveland Brothers, 911 and 913 Broadway, Albany, N. Y.]

Available carbonic acid	per cent..	12.58
Cubic inches per ounce of powder at 212°F		151.1

PERCENTAGE COMPOSITION.

Total carbonic acid, CO_2	13.21
Sodium oxide, Na_2O	13.58
Potassium oxide, K_2O	14.93
Calcium oxide, CaO18
Tartaric acid, $\text{C}_4\text{H}_4\text{O}_5$	41.60
Sulphuric acid, SO_310
Starch	7.42
Water of combination and association by difference	8.98
	<hr/> 100.00

PROBABLE PERCENTAGE COMBINATION.

Sodium bicarbonate, NaHCO_3	25.21
(Residual sodium oxide, Na_2O)	4.28
Potassium bitartrate, $\text{KHC}_4\text{H}_4\text{O}_6$	59.25
Calcium sulphate, CaSO_417
Starch	7.42
Water of association	3.67
	<hr/> 100.00

5507.—*Sea Foam (Gantz) Baking-Powder.*

[Made by Gantz, Jones & Co., 176 Duane street, New York.]

Available carbonic acid	per cent..	8.03
Cubic inches per ounce of powder at 212°F		96.5

PERCENTAGE COMPOSITION.

Total carbonic acid, CO_2	8.10
Sodium oxide, Na_2O	15.47
Potassium oxide, K_2O	15.24
Calcium oxide, CaO78
Tartaric acid, $\text{C}_4\text{H}_4\text{O}_6$	44.18
Starch	5.32
Water of combination and association by difference	10.91
	<hr/> 100.00

PROBABLE PERCENTAGE COMBINATION.

Sodium bicarbonate, NaHCO_3	15.46
(Residual sodium oxide, Na_2O)	9.77
Potassium bitartrate, $\text{KHC}_4\text{H}_4\text{O}_6$	62.92
(Calcium oxide, CaO)78
Starch	5.32
Water of association	5.75
	<hr/> 100.00

This and the preceding, No. 5505, contain very small quantities of starch, apparently rather too little for the proper preservation of the last sample.

5522.—*Hecker's Perfect Baking-Powder.*

[Made by George V. Hecker & Co., 205 Cherry street, New York.]

Available carbonic acid	per cent..	9.29
Cubic inches per ounce of powder at 212°F		111.6

PERCENTAGE COMPOSITION.

Total carbonic acid, CO_2	9.26
Sodium oxide, Na_2O	11.61
Potassium oxide, K_2O	11.63
Calcium oxide, CaO91
Tartaric acid, $\text{C}_4\text{H}_4\text{O}_6$	39.74
Sulphuric acid, SO_322
Starch	12.78
Water of combination and association by difference	13.85
	<hr/> 100.00

PROBABLE PERCENTAGE COMBINATION.

Sodium bicarbonate, NaHCO_3	17.67
(Residual sodium oxide, Na_2O)	5.09
Potassium bitartrate, $\text{KHC}_4\text{H}_4\text{O}_6$	56.60
Calcium sulphate, CaSO_437
(Calcium oxide, CaO)76
Starch	12.78
Water of association	6.73
	<hr/> 100.00

This sample contains rather more lime than most of the other tartrate powders. The excess given above is probably combined with tartaric acid as calcium tartrate; the same is true of the two following samples:

5527.—*Gilbert S. Graves's Imperial Baking-Powder.*

[Made by The Imperial Baking-Powder Company, Buffalo, N. Y.]

Available carbonic acid	7.28
Cubic inches per ounce of powder at 212°F	87.4

PERCENTAGE COMPOSITION.

Total carbonic acid, CO_2	8.47
Sodium oxide, Na_2O	13.62
Potassium oxide, K_2O	9.42
Calcium oxide, CaO45
Tartaric acid, $\text{C}_4\text{H}_4\text{O}_5$	32.74
Starch	24.57
Water of combination and association by difference	10.73
	<hr/> 100.00

PROBABLE PERCENTAGE COMBINATION.

Sodium bicarbonate, NaHCO_3	16.16
(Residual sodium oxide, Na_2O)	7.66
Potassium bitartrate, $\text{KHC}_4\text{H}_4\text{O}_6$	46.63
(Calcium oxide, CaO)45
Starch	24.57
Water of association	4.53
	<hr/> 100.00

5529.—*Thurber's Best Baking-Powder.*

[Made by H. K. & F. B. Thurber & Co., West Broadway, Reade and Hudson streets, New York.]

Available carbonic acid	per cent. 10.26
Cubic inches per ounce of powder at 212°F	123.2

PERCENTAGE COMPOSITION.

Total carbonic acid, CO_2	10.54
Sodium oxide, Na_2O	10.65
Potassium oxide, K_2O	12.26
Calcium oxide, CaO66
Tartaric acid, $\text{C}_4\text{H}_4\text{O}_5$	38.75
Sulphuric acid, SO_307
Starch	13.41
Water of combination and association by difference	13.66
	<hr/> 100.00

PROBABLE PERCENTAGE COMBINATION.

Sodium bicarbonate, NaHCO_3	20.12
(Residual sodium oxide, Na_2O)	3.22
Potassium bitartrate, $\text{KHC}_4\text{H}_4\text{O}_6$	55.19
Calcium sulphate, CaSO_412
(Calcium oxide, CaO)61
Starch	13.41
Water of association	7.33
	<hr/> 100.00

5513.—*Sterling Baking-Powder.*

[Made by Sterling Manufacturing Company, Baltimore, Md.]

Available carbonic acid	per cent..	9.53
Cubic inches per ounce of powder at 212°F		114
Leavening gas (available $\text{CO}_2 + \text{NH}_3$)	per cent..	9.90
Cubic inches per ounce of powder at 212°F		123.3

PERCENTAGE COMPOSITION.

Total carbonic acid, CO_2	10.66
Sodium oxide, Na_2O	10.38
Potassium oxide, K_2O66
Calcium oxide, CaO15
Ammonia, NH_337
Tartaric acid, $\text{C}_4\text{H}_4\text{O}_5$	21.94
Starch	40.05
Water of combination and association by difference	15.79
	<hr/> 100.00

PROBABLE PERCENTAGE COMBINATION.

Sodium bicarbonate, NaHCO_3	19.13
(Residual sodium oxide, Na_2O)	3.32
Ammonium bicarbonate, $\text{N}_3\text{H}_{11}\text{C}_2\text{O}_5$	1.14
Free tartaric acid, $\text{H}_2\text{C}_4\text{H}_4\text{O}_6$	24.93
Starch	40.05
Water of association	11.43
	<hr/> 100.00

This and the following are the only samples examined which were made up with *free* tartaric acid as the sole acid constituent. Both contain small quantities of ammonium bicarbonate. The small quantities of potassium and calcium oxide probably exist as tartrates.

5535.—*Our Best Baking-Powder.*

[Made by the Purity Chemical Works, Philadelphia, Pa.]

Available carbonic acid	per cent..	4.98
Cubic inches per ounce of powder at 212°F		9.8
Leavening gas (available $\text{CO}_2 + \text{NH}_3$)	per cent..	5.22
Cubic inches per ounce of powder at 212°F		65.5

PERCENTAGE COMPOSITION.

Total carbonic acid, CO_2	5.13
Sodium oxide, Na_2O	13.65
Calcium oxide, CaO07
Ammonia, NH_324
Tartaric acid, $\text{C}_4\text{H}_4\text{O}_5$	18.48
Sulphuric acid, SO_361
Starch	45.63
Water of combination and association by difference	16.19
	<hr/> 100.00

PROBABLE PERCENTAGE COMBINATION.

Sodium bicarbonate, NaHCO_3	9.01
(Residual sodium oxide, Na_2O)	10.32
Ammonium bicarbonate, $\text{N}_3\text{H}_{11}\text{C}_2\text{O}_5$74
Free tartaric acid, $\text{H}_2\text{C}_4\text{H}_4\text{O}_6$	21.00
(Sulphuric acid, SO_3)61
Starch	45.63
Water	12.69
	<hr/>
	100.00

The sulphuric acid in this sample probably is present as sodium sulphate, which may be simply an impurity in some of the salts.

PHOSPHATE POWDERS.

5506.—*Wheat Baking-Powder.*

[Made by Martin Kalbfleisch's Sons, New York.]

Available carbonic acid	per cent..	3.79
Cubic inches per ounce of powder at 212°F		45.5

PERCENTAGE COMPOSITION.

Total carbonic acid, CO_2	5.57
Sodium oxide, Na_2O	14.10
Potassium oxide, K_2O	7.49
Calcium oxide, CaO	11.96
Phosphoric acid, P_2O_5	36.69
Sulphuric acid, SO_316
Ammonia, NH_316
Water of combination and association by difference	23.87
	<hr/>
	100.00

This powder is quite peculiar in its make-up. It contains no starch or filling and has a very low per cent. of available gas. It contains a small quantity of ammonia bicarbonate. It contains a considerable amount of potassium oxide, which would indicate the presence of an acid phosphate of potash, as there is no other possible combination for this base, and the per cent. of lime is hardly sufficient to answer for all the phosphoric acid present.

5508.—*Rumford Yeast Powder.*

[Made by Rumford Chemical Works, Providence, R. I.]

Available carbonic acid	per cent..	12.86
Cubic inches per ounce of powder at 212°F		154.5

PERCENTAGE COMPOSITION.

Total carbonic acid, CO_2	13.47
Sodium oxide, Na_2O	12.66
Potassium oxide, K_2O31
Calcium oxide, CaO	10.27
Phosphoric acid, P_2O_5	21.83
Starch	26.41
Water of combination and association by difference	15.05
	<hr/>
	100.00

PROBABLE PERCENTAGE COMBINATION.

Sodium bicarbonate, NaHCO_3	25.71
(Residual sodium oxide, Na_2O)	3.17
Acid phosphate of lime, anhydrous:	
$\text{Ca}_3(\text{PO}_4)_2$	18.95
H_3PO_4	18.15
	<hr/>
	37.10
Starch	26.41
Water of association (phosphate)	7.61
	<hr/>
	100.00

5509. — *Horsford's Self-raising Bread Preparation.*

[Made by Rumford Chemical Works, Providence, R. I.]

THE ACID POWDER.

PERCENTAGE COMPOSITION.

Calcium oxide, CaO	16.78
Phosphoric acid, P_2O_5	23.97
Sulphuric acid, SO_3	6.00
Starch	20.81
Water of combination and association by difference	32.44
	<hr/>
	100.00
	<hr/>

PROBABLE PERCENTAGE COMBINATION.

Acid phosphate of lime:	
$\text{Ca}_3(\text{PO}_4)_2$	21.00
H_3PO_4	19.80
	<hr/>
	40.80
Calcium sulphate, CaSO_4	11.40
Starch	20.81
Water of association	26.99
	<hr/>
	100.00

This sample was different from all the others in being put up in two separate packages. One of these contained the acid ingredient, with starch to keep it from becoming dry, and the other, bicarbonate of soda. The directions are to mix the contents of both papers, if the whole is to be made use of at once, or to use two equal measures of the acid part to one of the soda. For analysis the contents of the papers were taken separately, and the results obtained from the acid part are given above. The other paper contained simply bicarbonate of soda of good strength.

For the determination of the available carbonic acid another sample was purchased, the entire contents of both papers thoroughly mixed, and a portion of the mixed powders weighed out and submitted to analysis. The first estimation made gave 13.56 per cent. available carbonic acid. By the time a duplicate estimation was made, perhaps an hour after the first, the per cent. had fallen to 12.03, showing a very rapid deterioration, or loss of available gas. Two subsequent estimations made the same day gave, respectively, 9.50 per cent. and 9. per

cent. Two determinations made the next day gave 8.35 per cent. and 8.35 per cent. This plan of keeping the acid and alkali ingredients separate appears to be an excellent one, though it is similar to the old method of using cream of tartar and baking-soda, with acid phosphate of lime substituted for the cream of tartar. The acid phosphate used in this sample contains considerable sulphate. The preceding sample (No. 5508), though made by the same firm, is entirely free from it, showing that it had been made from a better article of phosphate.

ALUM POWDERS.

5526.—*Vienna Baking-Powder.*

[Made by the Penn Chemical Works, Philadelphia, Pa.]

Available carbonic acid	per cent..	6.41
Cubic inches per ounce of powder at 212° F.....		77.0

PERCENTAGE COMPOSITION.

Total carbonic acid, CO ₂	7.90
Sodium oxide, Na ₂ O	6.99
Calcium oxide, CaO12
Aluminium oxide, Al ₂ O ₃	3.65
Ammonia, NH ₃	1.02
Sulphuric acid, SO ₃	10.11
Starch	45.41
Water of combination and association by difference	24.80
	<u>100.00</u>

PROBABLE PERCENTAGE COMBINATION.

Sodium bicarbonate, NaHCO ₃	18.98
(Residual sodium oxide, Na ₂ O)	1.43
Ammonia alum, anhydrous:	
Al ₂ (SO ₄) ₃	12.15
(NH ₄) ₂ SO ₄	2.65
NH ₃34
	<u>15.14</u>
Starch	45.41
Water of association and of crystallization (alum)	19.04
	<u>100.00</u>

5528.—*Metropolitan Baking-Powder.*

[Made by Metropolitan Perfume Company, Washington, D. C.]

Available carbonic acid	per cent..	8.10
Cubic inches per ounce of powder at 212° F.....		97.3

PERCENTAGE COMPOSITION.

Total carbonic acid, CO ₂	9.45
Sodium oxide, Na ₂ O	9.52
Aluminium oxide, Al ₂ O ₃	3.73
Ammonia, NH ₃	1.07
Sulphuric acid, SO ₃	10.71
Starch	43.25
Water of combination and association by difference	22.27
	<u>100.00</u>

PROBABLE PERCENTAGE COMBINATION.

Sodium bicarbonate, NaHCO_3	18.04
(Residual sodium oxide, Na_2O)	2.86
Ammonia alum, anhydrous:	
$\text{Al}_2(\text{SO}_4)_3$	12.42
$(\text{NH}_4)_2\text{SO}_4$	3.33
NH_321
	<hr/>
Starch	15.96
Water of association and crystallization (alum)	43.25
	<hr/>
	19.89
	<hr/>
	100.00

5531.—*Cottage Baking-Powder.*

[Made by New York Yeast Company, New York.]

Available carbonic acid	per cent..	6.62
Cubic inches per ounce of powder at 212°F		79.5

PERCENTAGE COMPOSITION.

Total carbonic acid, CO_2	7.80
Sodium oxide, Na_2O	6.77
Aluminium oxide, Al_2O_3	3.92
Ammonia, NH_394
Sulphuric acid, SO_3	10.63
Starch	52.29
Water of combination and association by difference	20.10
	<hr/>
	100.00

PROBABLE PERCENTAGE COMBINATION.

Sodium bicarbonate, NaHCO_3	14.89
(Residual sodium oxide, Na_2O)	1.28
Ammonia alum, anhydrous:	
$\text{Al}_2(\text{SO}_4)_3$	14.05
$(\text{NH}_4)_2\text{SO}_4$	3.47
NH_332
	<hr/>
Starch	17.84
Water of association and crystallization (alum)	52.29
	<hr/>
	13.70
	<hr/>
	100.00

MIXED POWDERS.

5514.—*Dooley's Baking-Powder.*

[Made by Dooley & Bro., New York.]

Available carbonic acid	per cent..	9.62
Cubic inches per ounce of powder at 212°F		115.6

PERCENTAGE COMPOSITION.

Total carbonic acid, CO_2	9.55
Sodium oxide, Na_2O	10.31
Potassium oxide, K_2O	4.51
Ammonia, NH_322
Aluminium oxide, Al_2O_3	3.25
Calcium oxide, CaO35
Sulphuric acid, SO_3	7.85
Tartaric acid, $\text{C}_4\text{H}_4\text{O}_5$	(?)
Starch.....	31.54
Water of combination and association (including tartaric acid) by difference.....	32.42
	<hr/> 100.00

In this and the following sample no satisfactory estimation of the tartaric acid could be obtained in presence of the alum. Both contain free tartaric acid, and the potash present is probably combined as bitartrate. This sample contains a trace of phosphoric acid. This form of mixed powder should undoubtedly be condemned.

5523.—*Miles' Premium Baking-Powder.*

[Made by Joseph H. Larzelere & Co., Philadelphia, Pa.]

Available carbonic acid.....	per cent..	3.56
Cubic inches per ounce of powder at 212°F		<u>42.8</u>

PERCENTAGE COMPOSITION.

Total carbonic acid, CO_2	3.43
Sodium oxide, Na_2O	10.05
Potassium oxide, K_2O	4.78
Calcium oxide, CaO28
Ammonia, NH_373
Aluminium oxide, Al_2O_3	3.59
Tartaric acid, $\text{C}_4\text{H}_4\text{O}_5$	(?)
Sulphuric acid, SO_3	9.65
Starch.....	18.72
Water of combination and association (including tartaric acid) by difference.....	48.77
	<hr/> 100.00

ALUM AND PHOSPHATE POWDERS.

5510.—*Henkels' Baking-Powder.*

[Made by Henkels Bros., Paterson, N. J.]

Available carbonic acid.....	per cent..	7.74
Cubic inches per ounce of powder at 212°F		<u>93.0</u>

PERCENTAGE COMPOSITION.

Total carbonic acid, CO_2	8.34
Sodium oxide, Na_2O	12.25
Aluminium oxide, Al_2O_3	3.57
Calcium oxide, CaO	1.80
Phosphoric acid, P_2O_5	5.60
Sulphuric acid, SO_3	9.79
Ammonia, NH_386
Starch	40.91
Water of combination and association by difference	16.88
	<hr/>
	100.00
	<hr/> <hr/>

PROBABLE PERCENTAGE COMBINATION.

Sodium bicarbonate, NaHCO_3	15.92
(Residual sodium oxide, Na_2O)	6.38
Ammonia alum, anhydrous:	
$\text{Al}_2(\text{SO}_4)_3$	11.89
$(\text{NH}_4)_2\text{SO}_4$	2.42
NH_324
	<hr/>
	14.55
Acid phosphate of lime, anhydrous:	
$\text{Ca}_3(\text{PO}_4)_2$	3.32
H_3PO_4	5.63
	<hr/>
	8.95
Starch	40.91
Water of association (phosphate) and crystallization (alum)	13.29
	<hr/>
	100.00

5511.—*Mason's Yeast-Powder.*

[Made by the Dixon Yeast-Powder Company, 231 Seventh street S. W., Washington, D. C.]

Available carbonic acid	per cent..	9.96
Cubic inches per ounce of powder at 212°F		119.6
		<hr/> <hr/>

PERCENTAGE COMPOSITION.

Total carbonic acid, CO_2	10.66
Sodium oxide, Na_2O	12.58
Aluminium oxide, Al_2O_3	4.27
Calcium oxide, CaO	1.17
Phosphoric acid, P_2O_5	3.58
Sulphuric acid, SO_3	11.02
Ammonia, NH_3	1.04
Starch	43.83
Water of combination and associations by differences.	11.85
	<hr/>
	100.00
	<hr/> <hr/>

PROBABLE PERCENTAGE COMBINATION.

Sodium bicarbonate, NaHCO_3	20.34
(Residual sodium oxide, Na_2O)	5.07
Ammonia alum, anhydrous:	
$\text{Al}_2(\text{SO}_4)_3$	14.22
$(\text{NH}_4)_2\text{SO}_4$	1.76
NH_359
	16.57
Acid phosphate of lime, anhydrous:	
$\text{Ca}_3(\text{PO}_4)_2$	2.16
H_3PO_4	3.57
	5.73
Starch	43.83
Water of association (phosphate) and crystallization (alum)	8.46
	100.00

5512.—Dixon Yeast-Powder.

[Made by the Dixon Yeast-Powder Company, 231 Seventh street. S. W., Washington, D. C.]

Available carbonic acid	per cent..	10.37
Cubic inches per ounce of powder at 212°F		124.6

PERCENTAGE COMPOSITION.

Total carbonic acid, CO_2	10.68
Sodium oxide, Na_2O	14.04
Calcium oxide, CaO	1.29
Aluminium oxide, Al_2O_3	4.59
Ammonia, NH_3	1.13
Phosphoric acid, P_2O_5	3.38
Sulphuric acid, SO_3	11.57
Starch	42.93
Water of combination and association by difference	10.39
	100.00

PROBABLE PERCENTAGE COMBINATION.

Sodium bicarbonate, NaHCO_3	20.39
(Residual sodium oxide, Na_2O)	6.52
Ammonia alum anhydrous:	
$\text{Al}_2(\text{SO}_4)_3$	15.28
$(\text{NH}_4)_2\text{SO}_4$	1.67
NH_376
	17.71
Acid phosphate of lime, anhydrous:	
$\text{Ca}_3(\text{PO}_4)_2$	2.38
H_3PO_4	3.16
	5.54
Starch	42.93
Water of association (phosphate) and crystallization (alum)	6.91
	100.00

This and the preceding sample are made by the same firm, and are very similar in composition.

5515.—*Patapsco Baking-Powder.*

[Made by Smith, Hanway & Co., Baltimore, Md. Put up in glasses.]

Available carbonic acid	per cent..	7.58
Cubic inches per ounce of powder at 212° F.....		91.1

PERCENTAGE COMPOSITION.

Total carbonic acid, CO_2	9.18
Sodium oxide, Na_2O	9.83
Aluminium oxide, Al_2O_3	4.55
Calcium oxide, CaO	2.77
Phosphoric acid, P_2O_5	1.44
Sulphuric acid, SO_3	13.01
Ammonia, NH_391
Starch.....	41.24
Water of combination and association by difference.....	17.07
	100.00

PROBABLE PERCENTAGE COMBINATION.

Sodium bicarbonate, NaHCO_3	17.52
(Residual sodium oxide Na_2O)	3.36
Ammonia alum, anhydrous:	
$\text{Al}_2(\text{SO}_4)_3$	15.15
$(\text{NH}_4)_2\text{SO}_4$	3.53
	18.68
Calcium sulphate, CaSO_446
Acid phosphate of lime, anhydrous:	
CaO	2.58
P_2O_5	1.44
	4.02
Starch.....	41.24
Water of association (phosphate) and crystallization (alum)	14.72
	100.00

This and the three samples following all have the same brand, but were put up in different shape, No. 5515 being contained in glass bottles, No. 5516 in tin cans, and No. 5517 being sold in bulk. No. 5519 is another brand made by the same firm. Of these, Nos. 5515 and 5517 give very similar results on analysis, while in Nos. 5516 and 5519 the numbers agree closely with one another, though quite different from the other two samples. In Nos. 5515 and 5517 the acid phosphate of lime is given simply as the sum of the per cents. of CaO and P_2O_5 found. There is probably considerable calcium sulphate in these samples, but if the sulphuric acid is combined with all the alum there is not enough left for combination with the lime.

5516.—*Patapsco Baking-Powder.*

[Made by Smith, Hanway & Co., Baltimore, Md. In tin cans.]

Available carbonic acid	per cent..	6.70
Cubic inches per ounce of powder at 212° F.....		55.16

PERCENTAGE COMPOSITION.

Total carbonic acid, CO ₂	7.62
Sodium oxide, Na ₂ O	9.21
Aluminium oxide, Al ₂ O ₃	3.61
Calcium oxide, CaO	2.66
Ammonia, NH ₃88
Phosphoric acid, P ₂ O ₅	3.28
Sulphuric acid, SO ₃	12.26
Starch.....	36.39
Water of combination and association by difference.....	24.09
	<hr/> 100.00 <hr/>

PROBABLE PERCENTAGE COMBINATION.

Sodium bicarbonate, NaHCO ₃	14.54
(Residual sodium oxide, Na ₂ O).....	3.84
Ammonia alum, anhydrous:	
Al ₂ (SO ₄) ₃	12.02
(NH ₄) ₂ SO ₄	3.41
	<hr/> 15.43
Calcium sulphate, CaSO ₄	3.02
Acid phosphate of lime, anhydrous:	
Ca ₃ (PO ₄) ₂	2.62
H ₃ PO ₄	2.87
	<hr/> 5.49
Starch.....	36.39
Water of association (phosphate) and crystallization (alum)	21.29
	<hr/> 100.00 <hr/>

5517.—*Patapsco Baking-Powder.*

[Made by Smith, Hanway & Co., Baltimore, Md. Sold in bulk.]

Available carbonic acid	per cent..	8.42
Cubic inches per ounce of powder at 212° F.....		101.1

PERCENTAGE COMPOSITION.

Total carbonic acid, CO ₂	9.55
Sodium oxide, Na ₂ O	8.33
Aluminium oxide, Al ₂ O ₃	4.72
Calcium oxide, CaO	2.66
Ammonia, NH ₃86
Phosphoric acid, P ₂ O ₅	1.78
Sulphuric acid, SO ₃	13.18
Starch	43.92
Water of combination and association by difference.....	15.00
	<hr/> 100.00 <hr/>

PROBABLE PERCENTAGE COMBINATION.

Sodium bicarbonate, NaHCO_3	18.21
(Residual sodium oxide, Na_2O)	1.60
Ammonia alum, anhydrous:	
$\text{Al}_2(\text{SO}_4)_3$	15.72
$(\text{NH}_4)_2\text{SO}_4$	3.34
	<hr/> 19.06
Acid phosphate of lime, anhydrous:	
CaO	2.66
P_2O_5	1.78
	<hr/> 4.44
Starch	43.92
Water of association (phosphate) and crystallization (alum)	12.77
	<hr/> 100.00

5519.—*Silver Spoon Baking-Powder.*

[Made by Smith, Hanway & Co., Baltimore, Md.]

Available carbonic acid	per cent..	7.33
Cubic inches per ounce of powder at 212°F		88.0

PERCENTAGE COMPOSITION.

Total carbonic acid, CO_2	8.26
Sodium oxide, Na_2O	7.26
Potassium oxide, K_2O76
Calcium oxide, CaO	2.29
Aluminium oxide, Al_2O_3	3.58
Ammonia, NH_391
Phosphoric acid, P_2O_5	3.61
Sulphuric acid, SO_3	8.78
Starch	41.26
Water of combination and association by difference	23.29
	<hr/> 100.00

PROBABLE PERCENTAGE COMBINATION.

Sodium bicarbonate, NaHCO_3	16.13
(Residual sodium oxide, Na_2O)	1.44
Ammonia alum, anhydrous:	
$\text{Al}_3(\text{SO}_4)$	11.92
$(\text{NH}_4)_2\text{SO}_4$72
NH_378
	<hr/> 13.42
Acid phosphate of lime, anhydrous:	
$\text{Ca}_3(\text{PO}_4)_2$	4.22
H_3PO_4	2.32
	<hr/> 6.54
Starch	41.26
Water of association (phosphate) and crystallization (alum)	21.21
	<hr/> 100.00

5520.—*Windsor Baking-Powder.*

[Made by Edwin J. Gillies & Co., 245 and 247 Washington street, New York.]

Available carbonic acid	per cent..	9.36
Cubic inches per ounce of powder at 212° F		<u>112.4</u>

PERCENTAGE COMPOSITION.

Total carbonic acid, CO ₂	9.86
Sodium oxide, Na ₂ O	11.92
Calcium oxide, CaO	1.76
Ammonia, NH ₃99
Aluminium oxide, Al ₂ O ₃	3.60
Phosphoric acid, P ₂ O ₅	5.01
Sulphuric acid, SO ₃	10.51
Starch	41.26
Water of combination and association by difference	15.09
	<u>100.00</u>

PROBABLE PERCENTAGE COMBINATION.

Sodium bicarbonate, NaHCO ₃	18.82
(Residual sodium oxide, Na ₂ O)	4.98
Ammonia alum, anhydrous:	
Al ₂ (SO ₄) ₃	11.99
(NH ₄) ₂ SO ₄	3.50
	<u>15.49</u>
Acid phosphate of lime, anhydrous:	
Ca ₃ (PO ₄) ₂	3.25
H ₃ PO ₄	4.86
	<u>8.11</u>
Starch	41.26
Water of association (phosphate) and crystallization (alum)	11.34
	<u>100.00</u>

5521.—*Davis' O. K. Baking-Powder.*

[Made by R. B. Davis, 112 Murray street, New York.]

Available carbonic acid	per cent..	8.10
Cubic inches per ounce of powder at 212° F		<u>97.3</u>

PERCENTAGE COMPOSITION.

Total carbonic acid, CO ₂	9.02
Sodium oxide, Na ₂ O	11.20
Calcium oxide, CaO	3.47
Ammonia, NH ₃	1.04
Aluminium oxide, Al ₂ O ₃	4.67
Phosphoric acid, P ₂ O ₅	8.95
Sulphuric acid, SO ₃	11.54
Starch	32.85
Water of combination and association by difference	17.26
	<u>100.00</u>

PROBABLE PERCENTAGE COMBINATION.

Sodium bicarbonate, NaHCO_3	17.22
(Residual sodium oxide, Na_2O)	4.85
Ammonia alum, anhydrous:	
$\text{Al}_2(\text{SO}_4)_3$	15.55
$(\text{NH}_4)_2\text{SO}_4$	1.09
NH_376
	<hr/> 17.40
Acid phosphate of lime, anhydrous:	
$\text{Ca}_3(\text{PO}_4)_2$	6.40
H_3PO_4	8.31
	<hr/> 14.71
Starch	32.85
Water of association (phosphate) and crystallization (alum)	12.97
	<hr/> 100.00

5524.—*Brunswick Yeast-Powder.*

[Manufactured in New York for M. & P. Metzger, 417 Seventh street, Washington, D. C.]

Available carbonic acid	per cent..	9.81
Cubic inches per ounce of powder at 212°F		117.8

PERCENTAGE COMPOSITION.

Total carbonic acid, CO_2	11.49
Sodium oxide, Na_2O	10.87
Calcium oxide, CaO	2.22
Aluminium oxide, Al_2O_3	3.35
Ammonia, NH_3	1.59
Phosphoric acid, P_2O_5	5.11
Sulphuric acid, SO_3	10.14
Starch	34.97
Water of combination and association by difference	20.26
	<hr/> 100.00

PROBABLE PERCENTAGE COMBINATION.

Sodium bicarbonate, NaHCO_3	21.93
(Residual sodium oxide, Na_2O)	2.78
Ammonia alum, anhydrous:	
$\text{Al}_2(\text{SO}_4)_3$	11.15
$(\text{NH}_4)_2\text{SO}_4$	3.86
NH_360
	<hr/> 15.61
Acid phosphate of lime, anhydrous:	
$\text{Ca}_3(\text{PO}_4)_2$	3.56
H_3PO_4	5.63
	<hr/> 9.19
Starch	34.97
Water of association (phosphate) and crystallization (alum)	15.52
	<hr/> 100.00

5525.—*The Atlantic and Pacific Baking-Powder.*

[Made by The Atlantic & Pacific Tea Company, New York.]

Available carbonic acid.....	per cent..	7.91
Cubic inches per ounce of powder at 212° F.....		95.0

PERCENTAGE COMPOSITION.

Total carbonic acid, CO ₂	9.45
Sodium oxide, Na ₂ O.....	12.15
Calcium oxide, CaO.....	1.93
Aluminium oxide, Al ₂ O ₃	3.25
Ammonia, NH ₃72
Phosphoric acid, P ₂ O ₅	5.71
Sulphuric acid, SO ₃	8.93
Starch.....	37.66
Water of combination and association by difference.....	20.20
	<hr/> 100.00 <hr/>

PROBABLE PERCENTAGE COMBINATION.

Sodium bicarbonate, NaHCO ₃	18.04
(Residual sodium oxide, Na ₂ O).....	5.15
Ammonia alum, anhydrous:	
Al ₂ (SO ₄) ₃	10.82
(NH ₄) ₂ SO ₄	2.41
	<hr/> 13.23 <hr/>
Acid phosphate of lime, anhydrous:	
Ca ₃ (PO ₄) ₂	3.56
H ₃ PO ₄	5.63
	<hr/> 9.19 <hr/>
Starch.....	37.66
Water of association (phosphate) and crystallization (alum).....	16.73
	<hr/> 100.00 <hr/>

5530.—*Silver King Baking-Powder.*

[Made by Shaw & Thomas, New York.]

Available carbonic acid.....	per cent..	4.99
Cubic inches per ounce of powder at 212° F.....		59.9

PERCENTAGE COMPOSITION.

Total carbonic acid, CO ₂	6.12
Sodium oxide, Na ₂ O.....	10.32
Calcium oxide, CaO.....	2.79
Ammonia, NH ₃	1.04
Aluminium oxide, Al ₂ O ₃	3.75
Phosphoric acid, P ₂ O ₅	5.89
Sulphuric acid, SO ₃	10.57
Starch.....	42.66
Water of combination and association by difference.....	16.86
	<hr/> 100.00 <hr/>

PROBABLE PERCENTAGE COMBINATION.

Sodium bicarbonate, NaHCO_3	11.68
(Residual sodium oxide, Na_2O)	6.01
Ammonia alum, anhydrous:	
$\text{Al}_2(\text{SO}_4)_3$	12.49
$(\text{NH}_4)_2\text{SO}_4$	3.02
NH_326
	<hr/> 15.77
Acid phosphate of lime, anhydrous:	
$\text{Ca}_3(\text{PO}_4)_2$	5.15
H_3PO_4	4.87
	<hr/> 10.02
Starch	42.66
Water of association (phosphate) and crystallization (alum)	13.86
	<hr/> 100.00

5532.—*Eureka Baking-Powder.*

[Made by G. S. Feeny, Wheeling, W. Va.]

Available carbonic acid	per cent..	7.62
Cubic inches per ounce of powder at 212°F		91.5

PERCENTAGE COMPOSITION.

Total carbonic acid, CO_2	9.57
Sodium oxide, Na_2O	11.36
Calcium oxide, CaO	1.93
Aluminium oxide, Al_2O_3	3.14
Ammonia, NH_385
Phosphoric acid, P_2O_5	2.66
Sulphuric acid, SO_3	11.30
Starch	44.32
Water of combination and association by difference	14.87
	<hr/> 100.00

PROBABLE PERCENTAGE COMBINATION.

Sodium bicarbonate, NaHCO_3	18.27
(Residual sodium oxide, Na_2O)	4.62
Ammonia alum, anhydrous:	
$\text{Al}_2(\text{SO}_4)_3$	10.45
$(\text{NH}_4)_2\text{SO}_4$	3.31
	<hr/> 13.76
Calcium sulphate, CaSO_4	3.38
Acid phosphate of lime, anhydrous:	
$\text{Ca}_3(\text{PO}_4)_2$99
H_3PO_4	2.92
	<hr/> 3.91
Starch	44.32
Water of association (phosphate) and crystallization (alum)	11.74
	<hr/> 100.00

5533.—*Silver Star Baking-Powder.*

[Made by E. Canby, Dayton, Ohio.]

Available carbonic acid.....	per cent..	7.61
Cubic inches per ounce of powder at 212° F.....		91.4

PERCENTAGE COMPOSITION.

Total carbonic acid, CO ₂	9.89
Sodium oxide, Na ₂ O.....	12.69
Potassium oxide, K ₂ O.....	.74
Calcium oxide, CaO.....	2.16
Aluminum oxide, Al ₂ O ₃	3.38
Ammonia, NH ₃77
Phosphoric acid, P ₂ O ₅	5.30
Sulphuric acid, SO ₃	10.66
Starch.....	37.57
Water of combination and association by difference.....	16.84
	100.00

PROBABLE PERCENTAGE COMBINATION.

Sodium bicarbonate, NaHCO ₃	18.88
(Residual sodium oxide, Na ₂ O).....	5.73
Ammonia alum, anhydrous:	
Al ₂ (SO ₄) ₃	11.25
(NH ₄) ₂ SO ₄	2.98
	14.23
Calcium sulphate, CaSO ₄	1.66
Acid phosphate of lime, anhydrous:	
Ca ₃ (PO ₄) ₂	2.73
H ₃ PO ₄	5.59
	8.32
Starch.....	37.57
Water of association (phosphate) and crystallization (alum).....	13.61
	100.00

5534.—*Purity Baking-Powder.*

[Made by Smith, Hanway & Co., Baltimore, Md.]

Available carbonic acid.....	per cent..	7.13
Cubic inches per ounce of powder at 212° F.....		85.6

PERCENTAGE COMPOSITION.

Total carbonic acid, CO ₂	9.32
Sodium oxide, Na ₂ O.....	12.29
Calcium oxide, CaO.....	4.55
Aluminium oxide, Al ₂ O ₃	3.34
Ammonia, NH ₃93
Phosphoric acid, P ₂ O ₅	3.53
Sulphuric acid, SO ₃	13.23
Starch.....	40.19
Water of combination and association by difference.....	12.62
	100.00

PROBABLE PERCENTAGE COMBINATION.

Sodium bicarbonate, NaHCO_3	18.75
(Residual sodium oxide, Na_2O)	5.37
Ammonia alum, anhydrous:	
$\text{Al}_2(\text{SO}_4)_3$	11.12
$(\text{NH}_4)_2\text{SO}_4$	3.59
	<hr/>
	14.71
Calcium sulphate, CaSO_4	5.58
Acid phosphate of lime, anhydrous:	
$\text{Ca}_3(\text{PO}_4)_2$	4.19
H_3PO_4	2.51
	<hr/>
	6.70
Starch	40.19
Water of association (phosphate) and crystallization (alum)	8.70
	<hr/>
	100.00

These analyses agree pretty closely, in a general way, with those made by Professors Weber and Cornwall, so far as their determinations go. Professor Cornwall's figures for carbonic acid are uniformly higher than mine, his method giving rather the total than the available per cent. of gas. It is evident that the determination of available carbonic acid made upon samples obtained in retail stores would vary more or less, according to the time that had elapsed since the sample was first put up. This is well shown by Professor Cornwall's determinations (page 586), made upon samples of the same brand of powder purchased at different times. It would manifestly be unjust, therefore, to decide arbitrarily that the relative values of the different brands were in the exact rank indicated by the results given in this determination. The best results in all the different investigations, however, are given by the tartrate and phosphate powders, the alum, and the alum and phosphate powders giving almost uniformly low percentages of carbonic acid. (There are several exceptions, however, notably Professor Cornwall's No. 36, "One Spoon" giving 16.77 per cent.¹ This shows the possibilities of an alum powder as regards its carbonic-acid strength.)

Professor Cornwall's average for twenty samples of alum and phosphate powders (no straight alum powders included) is 8.97 per cent.; for eight samples of tartrate powders, 11.60. Professor Weber's average for nineteen samples of alum powders is 7.58 per cent.; for eight samples of tartrate powders, 11.20 per cent. My average for twenty samples of both alum, and alum and phosphate powders is 8 per cent.; for eight samples of tartrate powders, 10.10 per cent. The only straight phosphate powders sold seem to be the various preparations made by the Rumford Chemical Works, and the "Wheat" powder; at least these are all obtained by any of the investigators. The carbonic acid strength of the former is uniformly good, slightly higher than the tartrate pow-

¹ Weber obtained only 5.75 per cent. from a powder with this brand in his investigation. Page—.

ders; the latter is a peculiar preparation, made up without any filling whatever, and gives a very low percentage of carbonic acid, except in one of Professor Cornwall's samples, which seems to have been obtained quite fresh.

FILLING.

It is evident that of several powders made up of the same materials, the one which contains the smallest proportion of inert matter or filling, other things being equal, will have the best carbonic-acid efficiency or "strength." On the other hand, if the amount used is too small for the proper preservation of the sample, it will deteriorate rapidly, and perhaps will show less strength after keeping a short time than other powders with a somewhat larger amount of filling. It becomes a question, therefore, as to the minimum limit of the amount of filling that is consistent with good keeping qualities. Professor Prescott¹ says on this point:

From 13 to 18 per cent. of starch is not too much for the permanence of a cream of tartar baking-powder, but filling beyond 20 per cent. must be held an unquestionable dilution.

In my samples, the average per cent. of starch in the bitartrate powders was 14.04; the highest was 24.57 per cent., and the lowest 5.32 per cent. The latter sample evidently did not contain enough, for it had a much lower carbonic-acid strength than most of those that had more filling. The bitartrate powder containing the maximum of filling, No. 5527, contained also the lowest per cent. of available carbonic acid. The powders made up with free tartaric acid contained much more filling, this being doubtless necessitated by the more hygroscopic character of the free acid. They contain, respectively, 40.05 and 45.63 per cent. of starch, and 9.53 and 4.98 per cent. of available carbonic-acid. Of the phosphate powders No. 5508 contains rather a large amount of filling, 26.41 per cent., while No. 5506 contains none at all, evidently to its detriment, as previously noted. Even the acid part of No. 5509 contains 20.81 per cent. of starch, although it is kept separate from the alkali. It is in the alum, and the alum and phosphate powders, however, that the highest percentages of filling are found. The average of all is 40.76 per cent. of starch, the maximum 52.29 per cent., the minimum, 31.54 per cent. Here we find the cause for the low per cents. of available carbonic acid in these powders, which should, theoretically, afford a higher carbonic-acid strength than any of the other classes. Whether a large amount of filling is more necessary where alum is used to prevent deterioration, whether it is added simply as a diluent, so that the amount of alum taken into the starch will be less apt to produce an injurious effect, or whether it is added to cheapen the powder, I can not say. The first hypothesis seems the most probable,

¹ Organic Analyses, 500.

especially if the alum is used with but a small proportion of its water of crystallization driven off. If the second is true, the object is not obtained, of course, for the more filling used the greater the quantity of powder required to produce the same aerating effect, and as for the third, alum and soda are about as cheap as starch.

It must be remembered that the percentages of starch given in the tables represent *anhydrous* starch.

"DOMESTIC BAKING-POWDERS."

It may be asked, can not the consumer make up his own baking-powders? The difficulties in the way of doing this may be enumerated as follows:

(1) The chemicals in the market, as purchased by the consumer, may not be pure, or of full strength, so that when combined in proper proportions they do not give good results.

(2) The proper proportions to use, and the necessity of thorough mixing to secure good results, would not be well understood by any one who was not a chemist.

(3) In order to prevent the action of the ingredients upon one another, and to preserve the strength of the powder unimpaired as long as possible, the manufacturer *dries* all his chemicals before mixing them, so as to drive off most of the adhering moisture. Baking-soda can not be dried much, as it loses its carbonic acid, and consequently its efficiency, at very low temperatures. The starch, however, containing as it does from 10 to 18 per cent. of moisture, can be thoroughly dried at 100° to 105° C., and its efficiency as a filling material greatly increased. The cream of tartar can also be thoroughly dried. This operation of drying chemicals at a temperature below that at which decomposition would occur seems rather too elaborate an operation for the kitchen.

These difficulties are more apparent than real, however. In answer to the first, it may be said that the bitartrate is the only chemical which is likely to be adulterated, and as there is no difficulty nowadays in obtaining a pure article in the wholesale market, it only requires the proper enforcement of adulteration laws to oblige the retailer to furnish a good article. The second objection may be met by furnishing the public simple formulæ for compounding such powders, and the third, which is doubtless the most serious, I believe can be overcome by using a larger proportion of filling, without drying the chemicals.

In the present days of cooking-schools, when so much interest is taken in the preparation of food, and in all branches of the culinary art, it may not be amiss to devote a little space to the discussion of this subject, although it is not, perhaps, strictly within the scope of the present investigation.

With a view of determining the possibility of making up baking-powders from a simple formula that could be used in the household, and

also to see what strength of powder could be obtained by lessening the quantity of filling used, I compounded a number of powders from commercial cream of tartar and soda, using different proportions of starch, and determined the per cent. of carbonic acid, both total and available, in each. The chemicals used were dried before mixing, and the latter operation very thoroughly performed.

Formula No. 1, containing 20 per cent. starch filling.

Cream of tartar.....	ounces..	8
Baking-soda.....	do....	4
Corn starch.....	do....	3
<hr/>		
Total carbonic acid	per cent..	13.39
Available carbonic acid.....	do....	11.96

Formula No. 2, containing about 15 per cent. starch filling.

Cream of tartar.....	ounces..	8
Baking-soda.....	do....	4
Corn starch	do....	2
<hr/>		
Total carbonic acid	per cent..	14.60
Available carbonic acid.....	do....	12.89

Formula No. 3, containing 10 per cent. starch filling.

Cream of tartar.....	ounces..	6
Baking-soda.....	do....	3
Corn starch	do....	1
<hr/>		
Total carbonic acid	per cent..	15.10
Available carbonic acid.....	do....	13.70

From the above it will be seen that most excellent results were obtained with these powders, made up by simple formulæ. The powder containing the least percentage of starch, Formula No. 3, gave 13.70 per cent. of available carbonic acid, nearly 1 per cent. more than the highest result obtained in any of the commercial samples. To be sure these powders were freshly made, and would doubtless deteriorate on keeping, those with the lowest amount of starch perhaps more rapidly than the others, as most of the commercial samples containing less than 10 per cent. of starch show low percentages of available carbonic acid, No. 5505 being an exception. But these prepared samples establish very completely the point I desired to make, that baking-powders can be readily made up by simple formulæ that will compare favorably with the best samples obtainable in the market.

These samples, however, were all made with *well-dried* ingredients, as they would be by a manufacturer. The next question is, whether a powder could be made which would keep without serious deterioration,

without drying the chemicals. To this end I used a larger proportion of starch according to the following formula :

Formula No. 4, made without drying the ingredients, containing 25 per cent. starch filling.

Cream of tartar	ounces..	8
Baking-soda	do....	4
Corn starch	do....	4
		<hr/>
Total carbonic acid	per cent..	12. 63
Available carbonic acid	do.....	10. 91

This gives a fairly good amount of available gas, considerably higher than the average of the commercial samples. Estimations of the available carbonic acid in the same sample after it had stood over two months in the laboratory showed absolutely no loss in strength. I had it tried in a practical way by several persons in the Department who used it in their kitchens, and reported excellent results, finding it fully as efficient in all respects as the powder they were accustomed to buy. The consumer can pay full retail price for the ingredients and still make it up for about half the price at which a good powder is sold, and if he makes sure of the quality of his cream of tartar he will have an article of which the purity is assured, and which has not lost in strength by being kept in stock an indefinite length of time by the retailer. I can see no reason why all housekeepers should not make their own baking-powder.

REGULATION OF THE SALE OF BAKING-POWDERS.

The best plan for the regulation by law of the sale of baking-powders in the present condition of our knowledge of their effect upon the system would seem to be to require the manufacturer to use a label giving approximately the composition, or analysis, of the powder sold. This is recommended by Professor Cornwall, and it appears to offer the best solution of the whole problem. The testimony that has been adduced is hardly sufficient to justify the prohibition of the sale of the cheaper kinds of powders as being injurious to health, but if they were required to be sold with a label giving their true composition it would soon lead to investigations upon this point. This is in harmony, also, with modern ideas in regard to legal regulation of the sale of food-stuffs, the tendency nowadays being to allow the sale of cheap substitutes for any article of food so long as they are not actually injurious to health, but to make all possible provision to insure that the purchaser should know exactly what he is getting, and that the substitute shall not be palmed off on him as the genuine article. In the case of baking-powders it is manifestly unjust to the public to allow the sale of a first-class tartrate powder and an alum powder as the same article, and it is equally unjust to the manufacturer of the higher-priced article. The nature of the sub-

stance is such that the purchaser has no means of ascertaining by any simple or easy means the character of the article he buys, to say nothing of its relative quality. Such a regulation should meet with the approbation of all concerned in the manufacture of baking-powders. The manufacturers of high-grade powders, such as tartrate or phosphate powders, would certainly not object to it, and it would ultimately be to the advantage also of the cheaper sorts, such as alum powders, provided they could succeed in proving that such powders produced little or no injury to the health of the consumer.

Ample analogy and precedent for such regulation are furnished by the laws for the sale of fertilizers which are in operation in most of the States. Although these substances are used for widely different purposes, the conditions that require the legal supervision of their sale are quite similar in many respects. A substance sold as a fertilizer must have its composition, in so far as is necessary for its valuation for such a purpose, plainly stated on the bag in which it is sold, because the purchaser has no means of ascertaining this value by any ordinary or simple test. Otherwise the manufacturer could easily impose upon him by selling him a powdered substance which resembled a fertilizer in general appearance, but contained no constituent of any value whatever for fertilizing purposes. The purchaser of a baking-powder receives a white powder which may contain various substances more or less valuable for the desired purpose, or of no value whatever, or perhaps even injurious to the health.

The housewife surely deserves protection against swindling as much as the farmer, and she has no better means for ascertaining the strength and quality of the baking-powder she buys than the latter has for learning the strength of his fertilizer. The verity and accuracy of the analysis stated on the label should be insured, as in the case of the fertilizer, by its being performed by sworn analysts. If such a regulation were enforced, people would soon inform themselves of the respective merits of different varieties, and the further requirement of a certain standard of strength, as suggested by Professor Cornwall, would probably be unnecessary, as they would learn to interpret the analysis, and a powder made up with 50 per cent. of starch, for instance, would have to be sold cheaper than one made with 10 per cent., or not sold at all.

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